

Report 10

TWO-STAGE SLURRY FISCHER-TROPSCH/ZSM-5 PROCESS OF
CONVERTING SYNGAS TO HIGH OCTANE GASOLINE

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TWO-STAGE SLURRY FISCHER-TROPSCH/ZSM-5 PROCESS
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I. Introduction

The title of the contract is, "Slurry Fischer-Tropsch/Mobil Two-Stage Process of Converting Syngas to High Octane Gasoline." This contract began on October 1, 1980 and will end on April 1, 1983. The overall objective of the contract is to develop a two-stage slurry F-T/ZSM-5 process for direct conversion of syngas to high octane gasoline (Slide #1). To achieve this objective, specific tasks are the design, construction, and operation of a two-stage bench-scale pilot plant, and the acquisition of necessary process information for the evaluation of the technical and economic potential of the process. In last year's project review meeting, this pilot plant was in the construction phase. This time, I am very happy to report the completion and smooth operation of the pilot plant.

In last year's meeting, I also took the opportunity to explain the reasons for which Mobil is undertaking the task of developing this two-stage synthesis gas conversion process concept. I am not going to repeat this here, except to mention that this process concept can directly convert low-cost low H₂/CO

ratio synthesis gas into high quality hydrocarbon fuels.

II. Present Status of the Project

Slide #2 summarizes the status of this project to date. The design and construction phases of the pilot plant are now complete. The pilot plant has also been successfully shaken down and is now fully operational. One slurry F-T catalyst, designated I-A (Fe/Cu/K₂CO₃), has been evaluated using the pilot plant. Currently, a second F-T catalyst, designated I-B (Fe/Cu/K₂CO₃), is being evaluated and has demonstrated high activity and stability over forty days. The operational results of the later run will be reported here. We also significantly improved the slurry F-T reactor mathematical model since my last report. Some details of this model and its application will also be reported here. My presentation today will follow the order given in this slide.

III. The Slurry Fischer-Tropsch/ZSM-5 Two-Stage Bench-Scale Pilot Plant

Slide #3 shows a simplified flow diagram of the bench-scale pilot plant. This same slide was shown in last year's meeting. Basically, the pilot plant is divided into four sections:

- I. Feed Gas
- II. First-Stage Slurry F-T Bubble-Column
- III. Second-Stage Fixed-Bed ZSM-5 Reactors (two in parallel) and Product Recovery

IV. Liquid Hydrocarbon Product Distillation

Slide #4 shows the physical appearances of the pilot plant. The tall white column embedded in a steel structure on the right-side of the picture is the 5.2 cm(D) x 760 cm(L) slurry F-T bubble-column reactor. The instrumental panel is on the left-side of the picture. The Gas Feed, Product Recovery, and Liquid Hydrocarbon Product Distillation Sections are in the middle. Two second-stage ZSM-5 reactors are on the left side of the steel structure housing the slurry bubble-column.

IV. A Slurry Fischer-Tropsch Reactor Mathematical Model

Since the slurry F-T reactor is rather unconventional and involves complicated transport phenomena, a mathematical model of such a system was developed to assist the design and the operation of the pilot plant slurry reactor. Since the beginning of this project, we have gone through several developmental stages for the mathematical model. In last year's meeting, a preliminary model and its application were reported. This time, I would like to report the latest version of the model. Slide #5 summarizes the major assumptions used in this latest model. The major improvements over the preliminary model that I presented last year are the following three changes:

1. A multiple-component system (H_2 , CO, CO_2 , H_2O) instead of a single-component system (H_2).
2. A non-linear F-T kinetic expression of Langmuir-Hinshelwood-type instead of a linear kinetic expression.
3. A water-gas shift reaction.

The new non-linear F-T kinetic expression is consistent with the hypothesis of competitive adsorption of active species on the same catalytic active-site. Only the [CO] and [H_2O] appearing in the denominator indicate that both species are strongly adsorbed on the catalyst active sites. This expression was first proposed by Dry (1976). The strongest implication of this expression on the model application is that the apparent kinetics approaches a second-order reaction when the CO concentration becomes small, while it maintains a first-order reaction when the CO concentration is large. The inclusion of the water-gas shift reaction enables the model to estimate the H_2/CO usage ratio instead of using a predetermined value. It has been shown that the H_2/CO usage ratio varies substantially over the reaction path. The kinetic expression for the water-gas shift reaction is consistent with the mass-action law with a denominator identical to that used in the F-T kinetic expression. The constant k_4 is the thermodynamic equilibrium constant for the water-gas shift reaction.

Slide #6 summarizes the material-balance equations based on the assumptions given in the previous slide. These equations are self-explanatory. Together with the inlet conditions, this set of non-linear equations is solved numerically using the orthogonal collocation method (Villadsen and Michelsen, 1978).

The physical parameters used in the mathematical model, such as the gas solubilities, the liquid-phase diffusivities, and others, were all obtained or estimated from published sources. However, the kinetic parameters (k_1 , k_2 , and k_3) were estimated based on a set of literature data obtained from a bench-scale F-T bubble-column reported by Koelbel and Ralek (1980). The best values of the parameters were estimated by using a method of parametric regression to minimize a predetermined target function.

This model, and the previous preliminary models, were used to assist the design and the operation of the pilot plant slurry F-T reactor. The major conclusions are summarized in slide #7. We have found that, in order to get high synthesis gas conversion at the design gas flow rate of the pilot plant F-T reactor, an active catalyst and a small gas-bubble size are essential. The axial liquid mixing in the long, slim pilot plant F-T reactor is small and has little effect on the reactor performance. The axial catalyst distribution depends strongly on the catalyst size. Its effect on reactor performance is small if

the catalyst size is less than 40 μm . The large effects due to both the superficial gas velocity and the catalyst loading are not unexpected since both directly affect the space velocity of the operation. I would like to examine the last conclusion more closely in the next two slides. These will show you, as examples, our efforts in this direction. As shown in Slide #8, the effect due to varying feed H_2/CO ratio on the pilot plant F-T reactor performance is rather small, except in the high synthesis gas conversion region. In that region, the use of a more stoichiometrically balanced H_2/CO feed gas may strongly increase the conversion. However, the catalyst is exposed to a very different atmosphere as indicated in Slide #9. The hydrogen-poor atmosphere associated with the low H_2/CO feed gas may significantly affect the aging characteristics of the catalyst. This has yet to be evaluated experimentally.

V. Pilot Plant Operation

Pilot plant construction was completed in late December, 1981. Its shakedown operation was carried out immediately afterward. The first trial-run of the pilot plant was initiated on March 16 this year. Since then, more than 120 days on-stream-time have been accumulated in the operation of the pilot plant. Slide #10 summarizes the major conclusions obtained from all the pilot plant runs. Currently, the pilot plant is in Run 3. The operation of the pilot plant has been very smooth. A high catalyst loading, together with long-term high synthesis gas

conversion at the designed high feed gas flow rate, has been achieved. We are currently evaluating an active gasoline-mode F-T catalyst. So far, more than forty days on-stream-time has been accumulated on this catalyst. The conversion of the F-T products into high octane gasoline using a second-stage ZSM-5 reactor has also been demonstrated. We are currently searching for a policy to maintain the optimum gasoline yield in the operation of the ZSM-5 reactor. It has also been observed that, when the methane and ethane yield is low, the yield of the reactor-wax (the heavy hydrocarbons retained in the reactor at the operating conditions) increases substantially. A reactor-wax yield of 33 wt % of the total hydrocarbon product with a 7 wt % yield of methane and ethane was observed. In the next few slides, some details of the pilot plant runs will be given.

Slide #11 illustrates the excellent temperature control exhibited by the slurry F-T reactor of the pilot plant. The circles are the temperature readings by the thermocouples located at the center of the bubble-column reactor. The temperature at the 8 cm location is purposefully maintained at 1-2°C lower by adjusting the feed-gas temperature. This is a precaution procedure that may not be necessary. The temperature reading at the 750 cm location is about 16°C below the expected slurry temperature when there is no slurry presented there. However, its reading will quickly reach the slurry temperature when the slurry reaches that location. We use this temperature change as

an additional indicator for the slurry level in the reactor. After some theoretical examination of this problem, we think that the actual temperature of the gas not far from the thermocouple shall be very close to the slurry temperature. The lower reading of the thermocouples results from a heat loss to the flange where a lower temperature is purposefully maintained. The two points in square are the inlet and exit temperatures of the Mobiltherm* flowing through the cooling jacket. They are about 5-6°C colder than the slurry temperature in order to remove the heat of the reactions. Also, the exit Mobiltherm temperature is usually 1-2°C higher than the inlet Mobiltherm temperature, the difference representing the reaction heat carried out by the Mobiltherm.

At the current stage, the operation of the second-stage ZSM-5 reactors is not a crucial matter, since we do not expect any major difficulties in their operation. However, we took this opportunity to search for a policy to operate the ZSM-5 reactor at a desired but constant severity of the catalytic activity. Currently, we try to use the inlet temperature as the parameter to control the catalytic severity. Slide #12 shows two temperature profiles in a fixed-bed ZSM-5 reactor (TOS = 579 and 777 hours, respectively). The catalyst severities at these two times were approximately the same. The higher inlet temperature required for the 777 hours TOS was to compensate the catalyst aging. The yardstick used to monitor the catalyst severity in

this case was the $i\text{-C}_4/(\text{C}_3^{\text{m}}+\text{C}_4^{\text{m}})$ ratio in the off-gas from a downstream separator. The catalyst temperature increases shown here are 45-50°C, very close to what we expected to be the adiabatic temperature increase.

Slide #13 summarizes the latest run of the pilot plant (CT-256-3) up to the end of last month. The run was being continued at that time. The results shown here demonstrate a very stable operation with high catalyst loading, high synthesis gas throughput, and high synthesis gas conversion. The F-T catalyst pretreatment was carried out at a temperature about 20°C higher than the synthesis temperature and was completed within 9.5 hours. The synthesis operation was very stable and smooth except for two minor emergency shutdowns, one caused by a false alarm and the other by leaks at the flange 305 cm above the feed-gas distributor. In both emergency shutdown cases, the reactor was immediately purged with nitrogen and brought back to synthesis operation as soon as the emergency problems were resolved. However, in both cases a slight drop in the synthesis gas conversion was observed after the restartup. To restore the synthesis gas conversion to the same level as before the shutdowns, the feed-gas velocity was decreased by 4% in both cases. Otherwise, this run was uneventful and the catalyst showed great stability. The operation of the second-stage reactor followed closely with the first-stage reactor operation. To compensate for the aging of the ZSM-5 catalyst, it was found

that a daily increase of the reactor inlet temperature by 4-6°C was needed.

Slide #14 illustrates the pretreatment of a F-T catalyst, designated I-A (Fe/Cu/K₂CO₃), in the pilot plant slurry reactor. The variables that were monitored during this pretreatment were synthesis gas contraction and conversion, and the CO₂ and methane concentrations in the off-gas from a downstream separator. Except for the initial transient period of the run, the run was very steady up to the sixteenth day, when the unit was shut down due to a false alarm. When the unit was back on normal operation, it took more than one day for the conversion to become stabilized. At that time, the feed-gas flow rate was decreased by about 4% in order to maintain the same conversion level as that before the shutdown. In this plot, the triangular points represent the data obtained with inter-reactor sampling when the second-stage reactor was in operation. One can conclude that the second-stage reactor contributed nothing to the synthesis gas conversion and insignificantly to the formation of the methane and ethane. There was practically little change of the ethane yield, but a slight drifting upward of the methane yield with increasing time-on-stream. There was definitely a step increase of the methane yield after the emergency shutdown.

As far as the bench-scale slurry bubble-column operation is concerned, there is a set of yardstick data that people often use for comparison. That is the data from a bench-scale unit reported by Koelbel and Ralek (1980). These data are given in Slide #16 and are compared against our current run CT-256-3. Two runs have almost identical space velocities, although our run was operated at a somewhat higher pressure. However, the temperature of our run is 6°C lower. In terms of the conversion, we have a slightly higher CO conversion and a somewhat lower H₂ conversion. This may indicate that our catalyst has a relatively higher water-gas shift activity than the Koelbel's catalyst has. In general, these two runs are very similar except for their methane + ethane yield, and their reactor-wax selectivities. A comparison of these selectivities will require further studies. We were never able to generate the Koelbel's low methane + ethane yield with our catalyst I-B. We were also very surprised by our large reactor-wax yield since Koelbel never reported any reactor-wax formation during the gasoline-mode operation. I strongly suspect that when we are able to substantially lower the methane and ethane yield, then the reactor-wax yield will be drastically increased as consistent to the Schulz-Floxy distribution of the F-T hydrocarbon products.

The performance of the second-stage ZSM-5 reactor is illustrated in Slide #17. The first column shows the product yields before the ZSM-5 reactor, while the second column shows

those after the ZSM-5 reactor. This comparison clearly shows the functions of the ZSM-5 catalyst, as given in the following:

- Conversion of light olefins to heavier hydrocarbons.
- Conversion of heavy hydrocarbons to lighter hydrocarbons.
- Formation of a large quantity of aromatics.
- Formation of large quantities of propane and i-butenes.

The potential total gasoline and heavier hydrocarbon yield after the alkylation of the i-butenes and light olefins is about 80 wt %. Additional gasoline can be obtained by converting the remaining light olefins to gasoline fraction. This yield is given as an example only. Further optimization of the liquid fuel yield shall be performed in the future.

The raw liquid hydrocarbons collected after the second-stage ZSM-5 reactor were analyzed to evaluate its quality as a gasoline fraction product. The properties of a typical product are given in Slide #18. Not surprisingly, it has a high octane number. The ASTM distillation curve seems to be reasonable except for the endpoint and the small amount of residue which are slightly higher than the specification of conventional gasoline.

The properties given in Slide #17, however, do not represent those of the total final gasoline. In a commercial plant, the total gasoline would be consisted of three major sources; i.e., the liquid hydrocarbons collected from the product separators, the C_4^+ hydrocarbons collected from gaseous product stream of the product separators, and the alkylate obtained from the alkylation of the i-butane and the propylene and butenes in the final product.

VI. Future Work

My last slide (Slide #19) shows the remaining work for this Contract. We would like to evaluate a low methane/ethane F-T catalyst. From the process economic point of view, this catalyst makes a lot of sense. However, we need to quantify its reactor-wax yield, activity, and stability. We also plan to run a long-term aging run of a gasoline-mode operation. Finally, we will perform a scoping-type process economic study using the design data obtained from our current operation.

Appendix A - Nomenclature

a_g	Gas bubble interfacial area, $6\epsilon_g/d_B$. (cm^2 gas-liquid area/mL expanded slurry)
C	Concentration, (mol/mL liquid or gas)
d_B	Bubble diameter, (cm)
K	Solubility coefficient, (mL liquid/mL gas)
k_1, k_2	Intrinsic kinetic rate constants for F-T and water-gas shift reactions, respectively, (mL liquid/s-gFe)
k_3, k_4	Constants used in the rate expressions for F-T and water-gas shift reactions
k_2	Liquid side mass transfer coefficient, (mL liquid/s- cm^2 gas-liquid area)
L	Bubble-column height, (cm)
m	Average H/C atomic ratio of F-T products
n	Average carbon-number of F-T products
r_1	Kinetic rate of F-T reaction, (mol/s-gFe)
r_2	Kinetic rate of water-gas shift reaction, (mol/s-gFe)
S_{ij}	Elements of stoichiometric matrix, $i = 1, \dots, 4$; and $j = 1, 2$
u	Superficial velocity, (cm/s)
v	Volumetric fraction of catalysts in slurry, $\rho_l w / (\rho_s + w(\rho_l - \rho_s))$, (mL catalyst/mL slurry)
w	Weight fraction of catalysts in slurry, (gCat/g slurry)
ϵ_g	<u>Greek Letters</u> Gas hold-up, (mL gas/mL expanded slurry)
ρ	Density, (g/mL)
ρ_s	Catalyst solid density, (g/mL)

Superscripts

i At reactor inlet

Subscripts

g Gas

i Components, $i = 1, 2, 3, 4$ for H_2 , CO, CO_2 , H_2O respectively

l Liquid

Appendix B - Literature

1. Dry, M. A., Ind. Eng. Chem., Prod. Res. Dev., 15(4), 282 (1976).
2. Koelbel, H., and Ralek, M., Cat. Rev. - Sci. Eng., 21, 225 (1980).
3. Villadsen, J., and Michelsen, M. L., "Solution of Differential Equation Models by Polynomial Approximations," Prentice Hall, Englewood Cliffs, NJ (1978).

SLIDE #1

CONTRACT TITLE

Slurry Fischer-Tropsch / Mobil Two-Stage Process of Converting Syngas to High Octane Gasoline (DE-AC22-80 PC 30022).

Contract Period: October 1, 1980 - April 1, 1983.

OVERALL OBJECTIVE

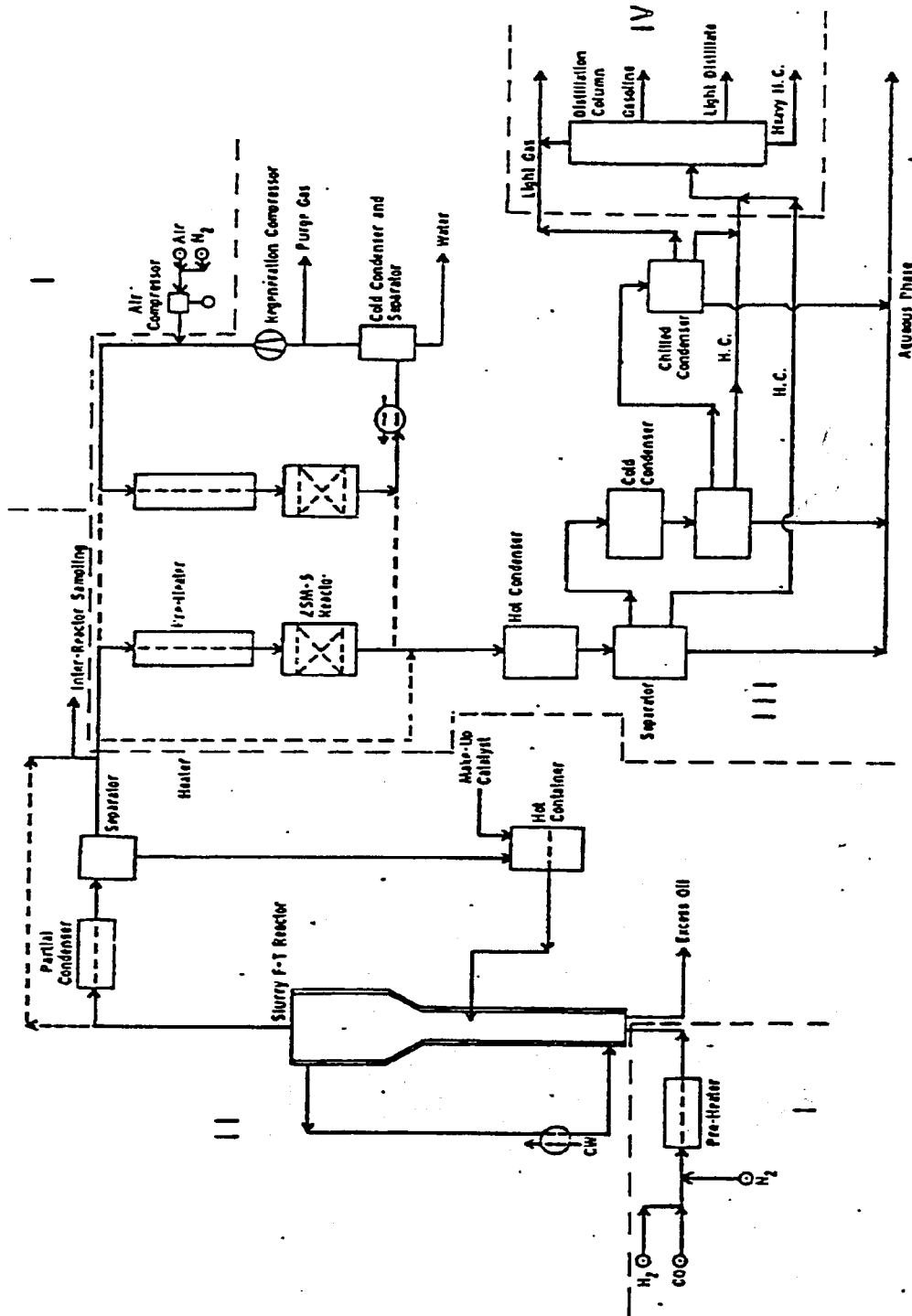
To Develop a Two-Stage Slurry Fischer-Tropsch / ZSM-5 Process for Direct Conversion of Syngas to High Octane Gasoline.

SLIDE #2

STATUS OF PROJECT

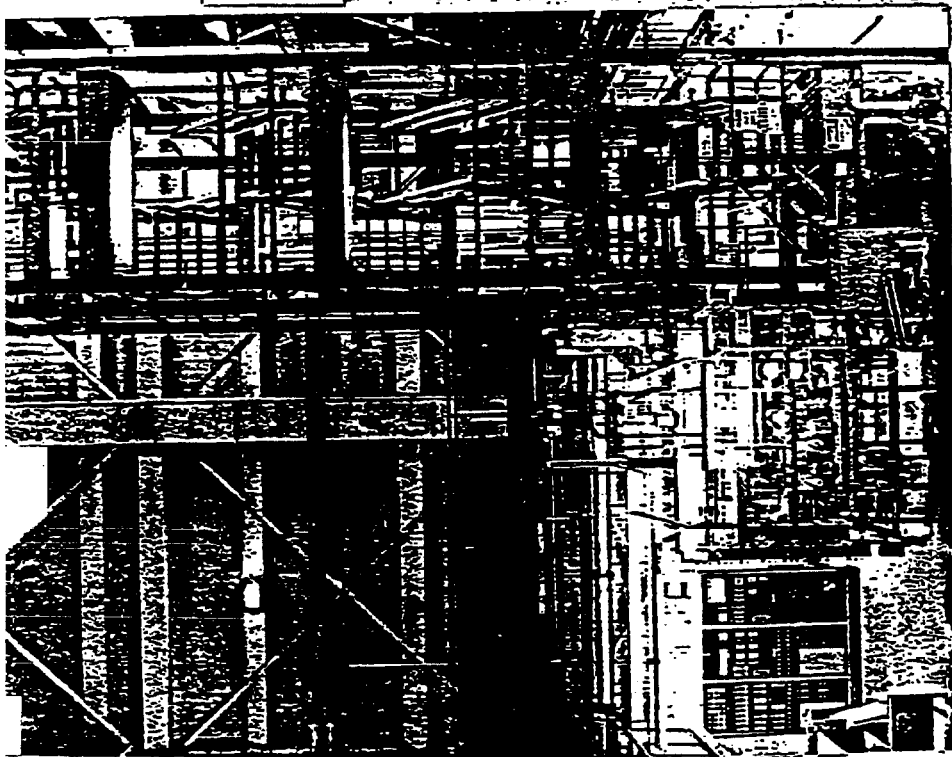
- The Design, Construction, and Shakedown of the Pilot Plant are Complete.
- A Slurry Fischer-Tropsch Reactor Mathematical Model Was Completed and Used to Assist the Design and Operation of the BSU F-T Reactor.
- A Slurry F-T Catalyst, Designated I-B (Fe / Cu / K_2CO_3), is Currently Under Evaluation. It is Highly Active and Has Accumulated More Than Thirty-Five Days Time-On-Stream.

SIMPLIFIED FLOW DIAGRAM OF TWO-STAGE PILOT PLANT FOR SYNTHESIS GAS CONVERSION



SLIDE #3

A North-west View of the Pilot Plant
— Control Panel (Left Bottom)
— Slurry Reactor "With White Insulation" (Right)
— Other Three Sections (Middle Bottom)



SLIDE # 4

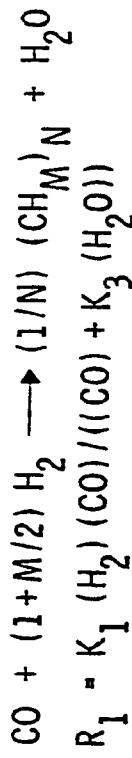
SIMPLIFIED FLOW DIAGRAM OF TWO-STAGE PILOT PLANT FOR SYNTHESIS GAS CONVERSION

MAJOR ASSUMPTIONS OF THE
LATEST F-T REACTOR MATHEMATICAL MODEL

1. Plug-Flow Gas and Non-Mixing Liquid-Phase
2. Uniform Catalyst Distribution in Liquid-Phase
3. Mass-Transfer Resistance Only at the Liquid Side of the Gas-Liquid Interface

4. Constant Molar Contraction Due to F-T Reactions

5. A Single Fischer-Tropsch Reaction:



6. A Water-Gas Shift Reaction:



7. A Steady-State and Isothermal Operation

SLIDE # 5

SLIDE #6

F-T REACTOR MATHEMATICAL MODEL EQUATIONS

$$- d(u_g C_{gi}) / dz$$

Convection in Gas Phase

$$= k_{gi} a_g (C_{gi} / K_1 - C_l)$$

Diffusion From Gas-Liquid Interface
to Liquid

$$= -(1 - \epsilon_g) (1 - v) C_{Fe} \sum_{j=1}^2 S_{ij} r_j$$

Kinetic Dissipation at Catalyst Surface

For $i=1, 2, 3, 4$ (H_2, CO, CO_2, H_2O)

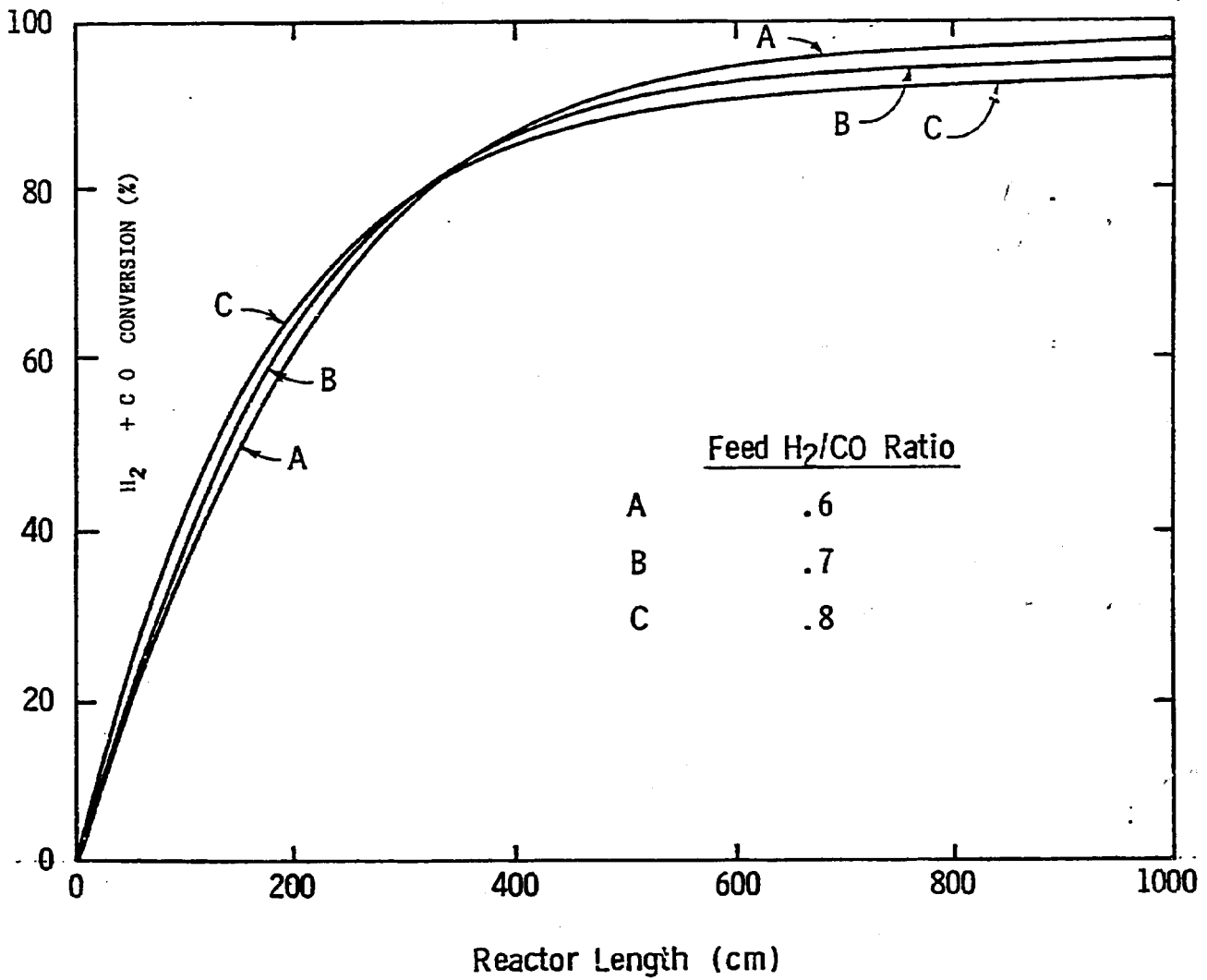
PILOT PLANT F-T REACTOR EVALUATION USING A
MATHEMATICAL MODEL

- An Active Catalyst and a Small Gas-Bubble Size Are Essential
- Axial Liquid Mixing Has Little Effect
- The Effect of the Axial Catalyst Distribution is Small if the Catalyst Size is Less Than 40 μm
- Both the Superficial Gas Velocity and the Fe-Catalyst Loading Have Large Effects
- The Feed H_2 / CO Ratio Has a Small Effect Except in the High Syngas Conversion Region

SLIDE # 7

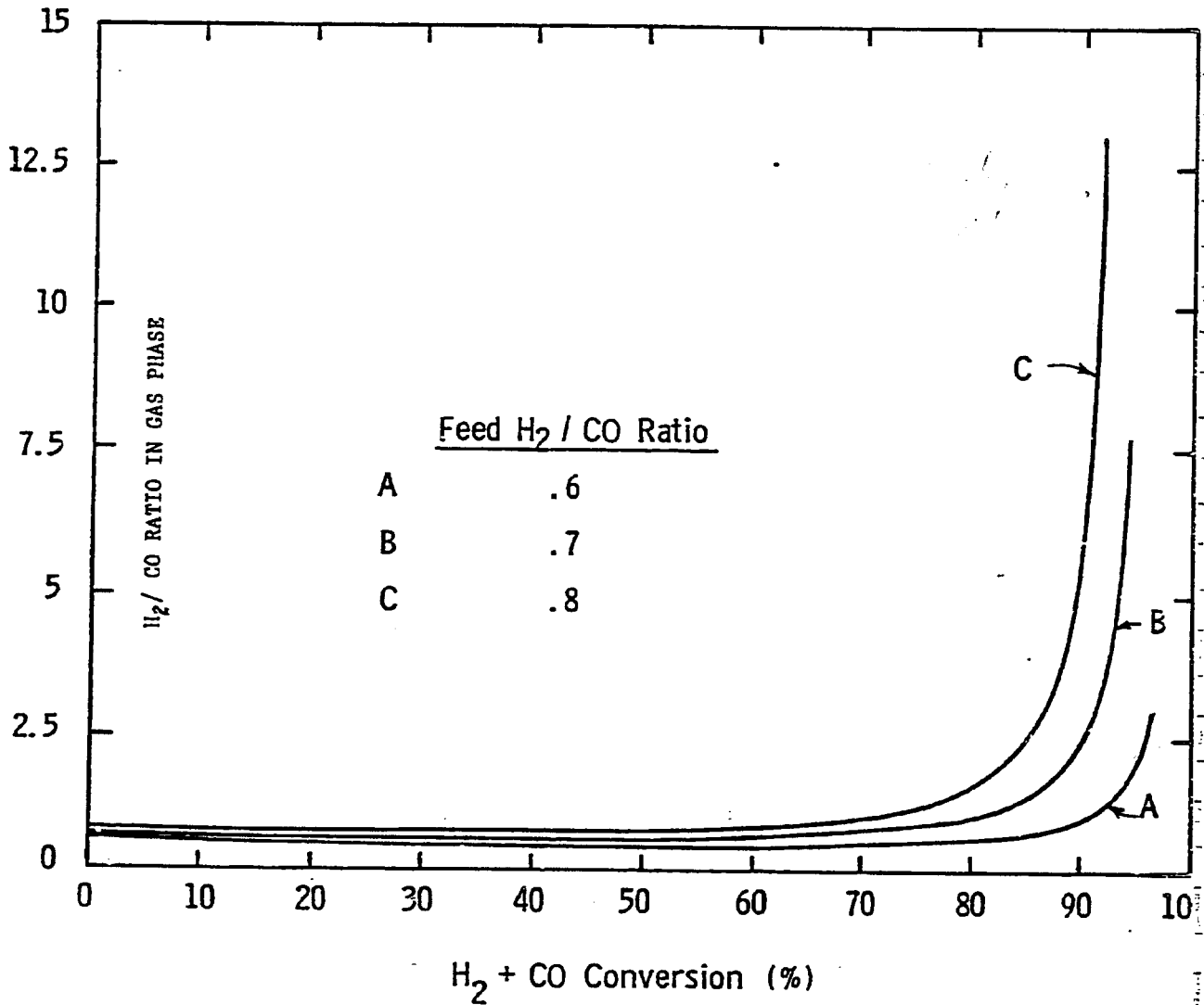
SLIDE #8

PREDICTED EFFECT OF FEED H₂/CO RATIO ON
H₂ + CO CONVERSION



SLIDE #9

PREDICTED EFFECT OF FEED H_2 / CO RATIO ON
 H_2 / CO RATIO IN GAS PHASE



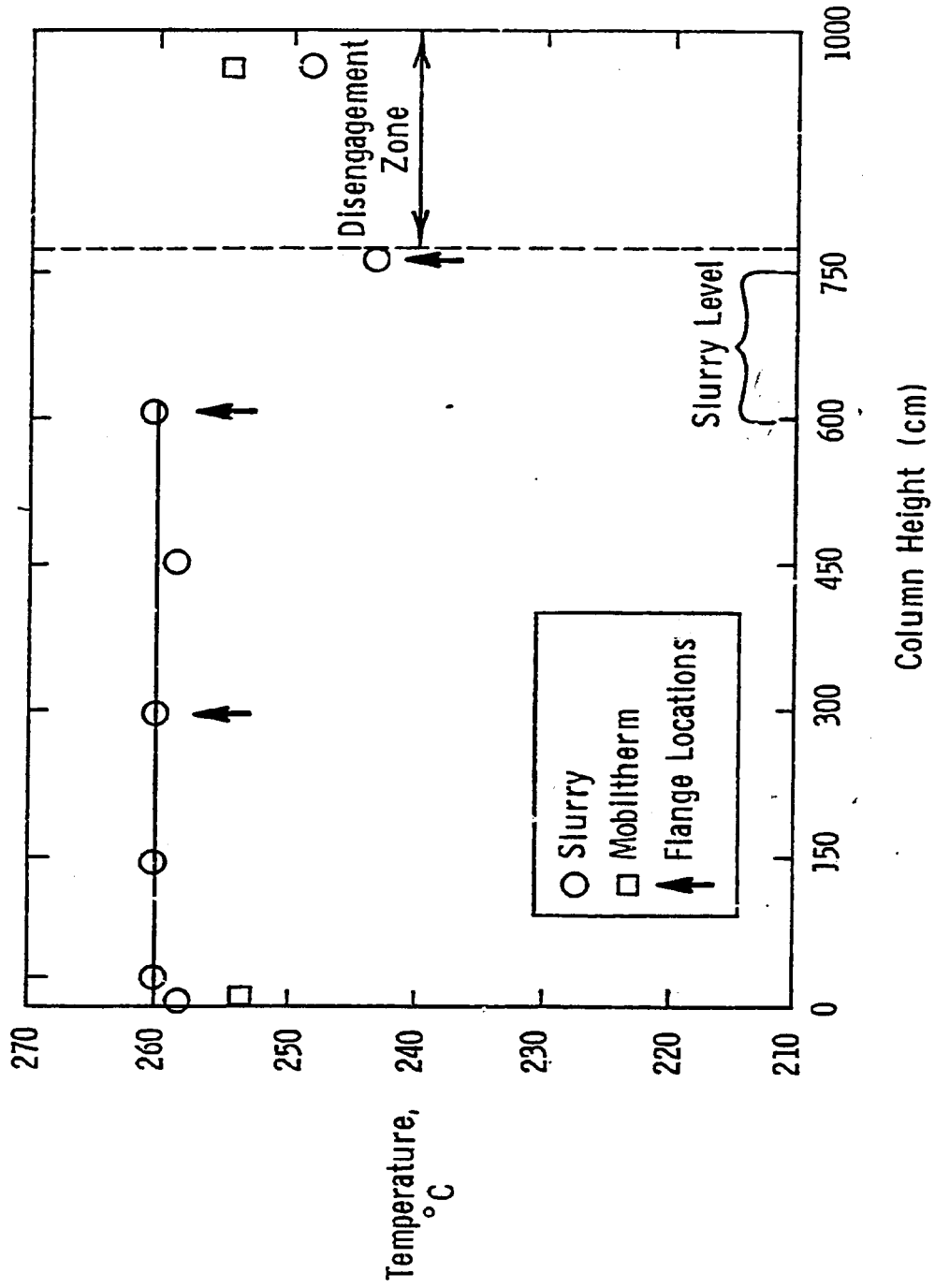
SLIDE #10

MAJOR CONCLUSIONS FROM PILOT PLANT RUNS

- Smooth Pilot Plant Operation Was Demonstrated
- High Catalyst Loading, Together With Long-Term High Syngas Conversion at the Designed High Syngas Flow Rate, Was Achieved
- A Stable and Highly Active Gasoline-Mode F-T Catalyst Is Being Evaluated
- The Conversion of the F-T Products Into High Octane Gasoline Was Demonstrated
- High Reactor-Wax Accumulation in the F-T Reactor is Expected When the Methane and Ethane Yield Is Low

SLIDE #11

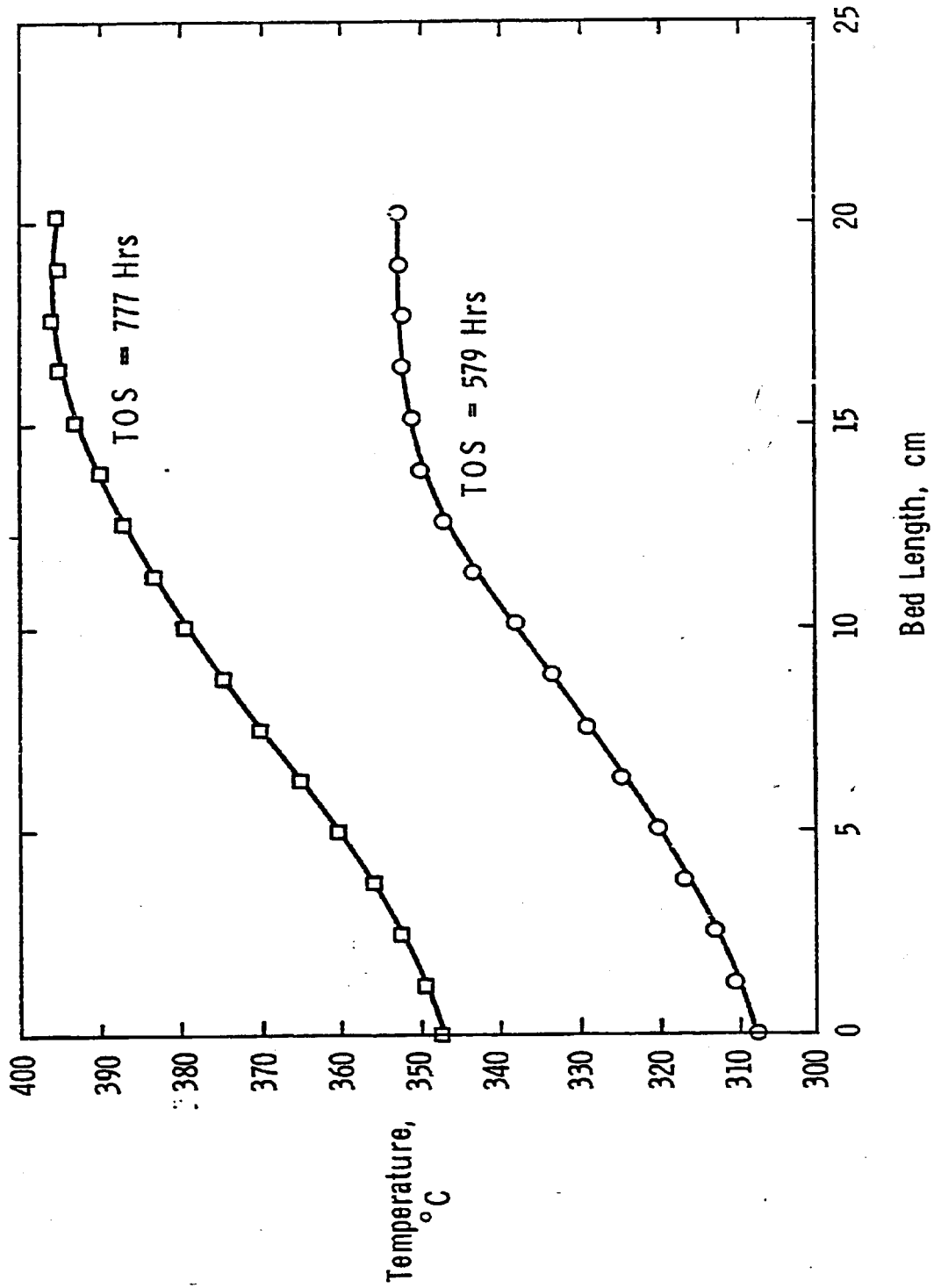
SLURRY BUBBLE COLUMN TEMPERATURE PROFILE
(Run CT-256-3 Time-On-Stream = 579 Hrs)



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SECOND-STAGE FIXED-BED ZSM-5 REACTOR
TEMPERATURE PROFILE
(Run CT-256-3)

SLIDE # 12



SLIDE #13
CT-256-3 RUN

Slurry Fischer-Tropsch Bubble-Column

Catalyst: I-B, Fe / Cu / K₂CO₃

Pretreatment:

Temperature, °C	282
Pressure, MPa	1.14
H ₂ / CO Molar Ratio	0.7
Space Velocity, NL / gFe-Hr	2.4
Total Time-On-Stream, Hrs	9.5

Synthesis:

Catalyst Loading, gms	1407
Temperature, °C	260 - 261
Pressure, MPa	1.14 - 1.48
H ₂ / CO Molar Ratio	0.7
Space Velocity, NL / gFe-Hr	2.4 - 2.9
Total Time-On-Stream, Hrs	800+
H ₂ + CO Conversion, Mol %	84 - 87* Excluding Transient Periods
Methane / Ethane Yield, Wt %	5.9 - 8.5 / 2.6 - 3.3

Fixed-Bed ZSM-5 Reactor

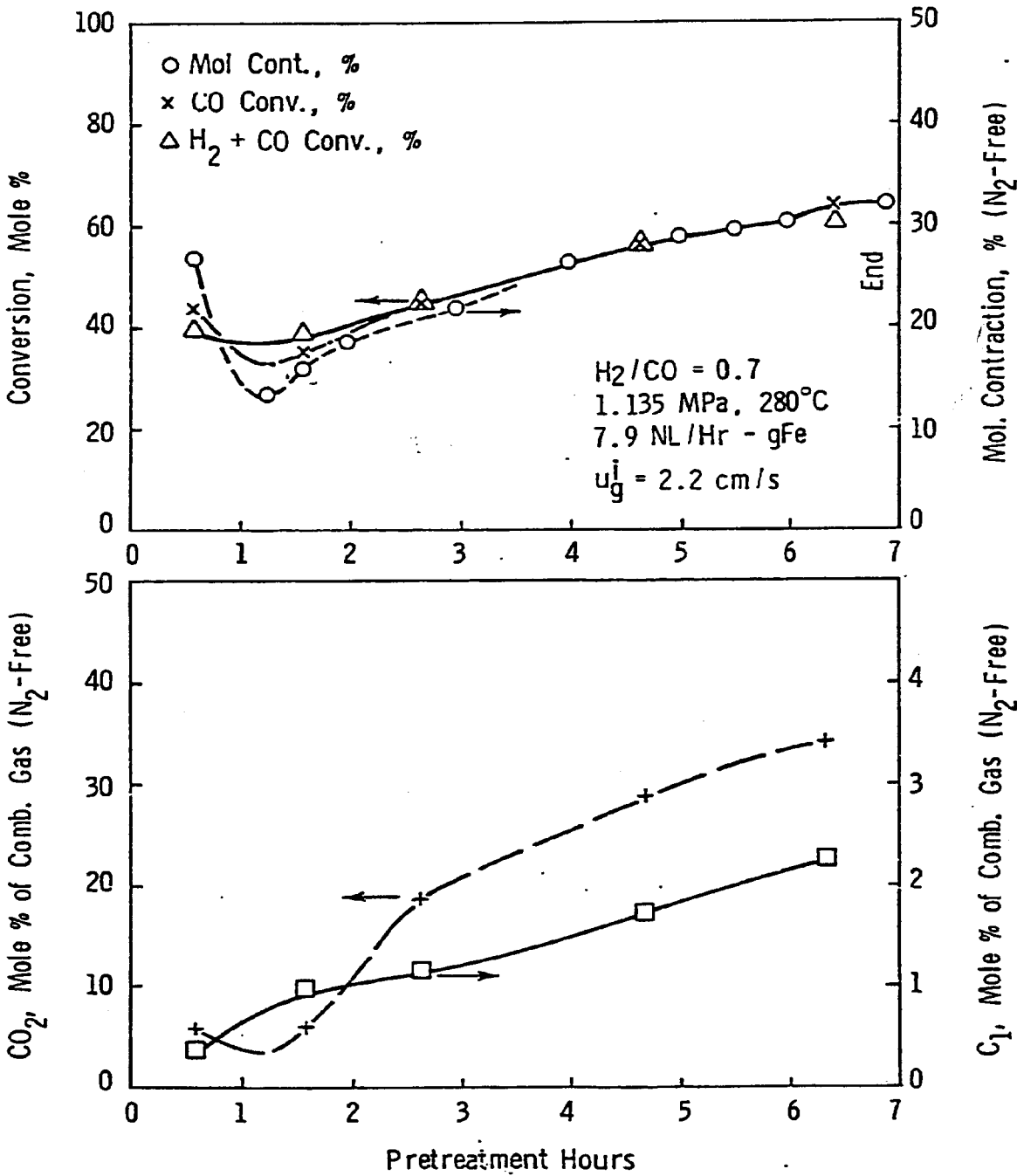
Catalyst: II-B, ZSM-5

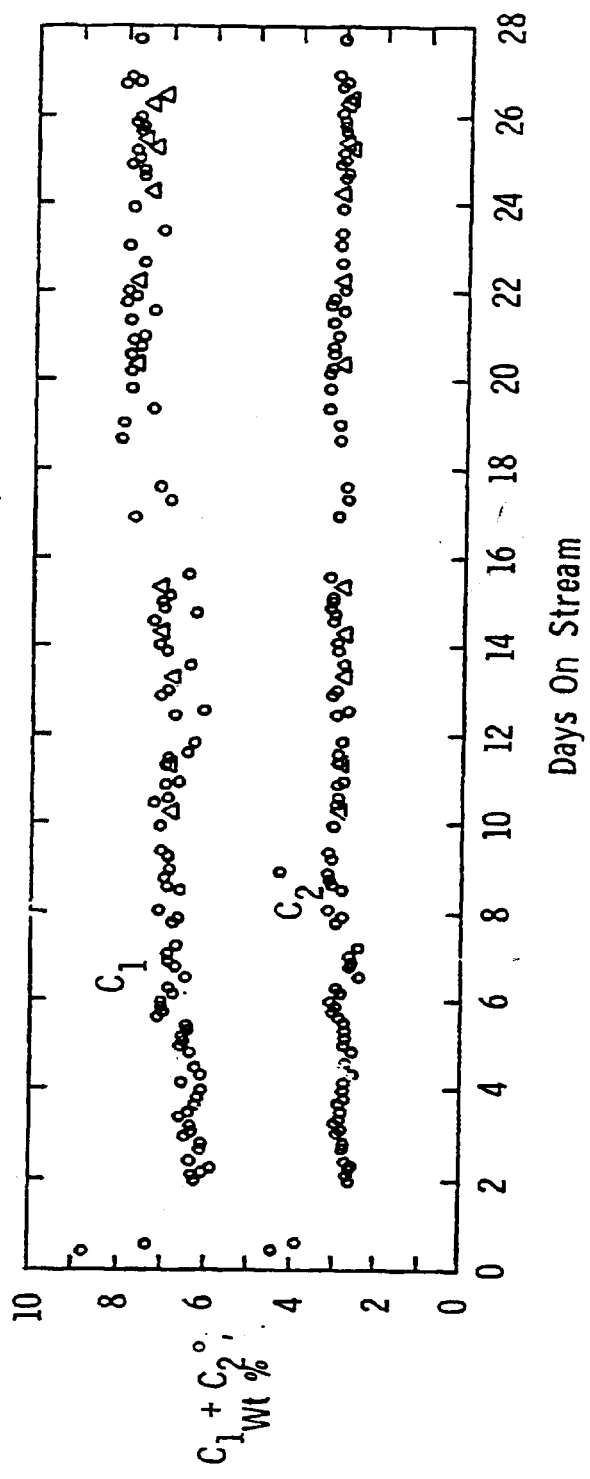
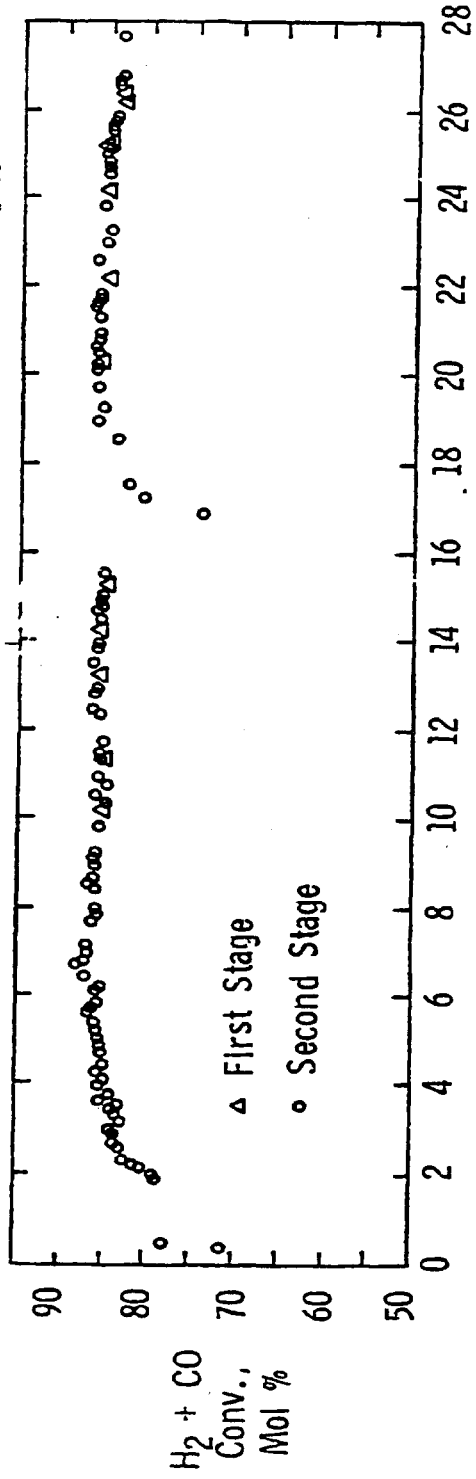
Reaction:

Inlet Temperature, °C	304 - 347+
Outlet Temperature, °C	353 - 396
Pressure	Cascaded
GHSV (STP), 1 / Hr	2265 - 2875
Total Time-On-Stream, Hrs.	550+

SLIDE #14

RUN CT-256-1, PRETREATMENT OF F-T CATALYST I-A
 (CATALYST I-A: Ppted. Fe / Cu / K_2CO_3)





SLIDE # 16
 COMPARISON OF RUN CT-256-3 VERSUS
 KOELBEL'S BENCH-SCALE PILOT PLANT RUN

	CT-256-3 (222 Hrs TOS)	Koelbel and Ralek*
Reactor D _{xL} , cm	5.2 x 730	4.7 x 350
Temperature, °C	260	266
Pressure, MPa	1.5	1.1
Sup. Gas Vel., cm / s	3.9	3.5
Feed H ₂ / CO	0.67	0.67
Cat. Loading, Wt %	14.3	17.2 (Est.)
Space Vel., NL / gFe - Hr	2.80	3.25
Conversion, Mol %		
H ₂	80	85
CO	91	90
H ₂ + CO	86	88
Selectivity, Wt %		
C ₁ + C ₂ ^o	10	3
Reactor-Wax	11	-

* Koelbel and Ralek, Cat. Rev. - Sci. Eng., 21, 225-274 (1980).

HYDROCARBON SELECTIVITIES

Before and After ZSM-5 Reactor
(Run CT 256-3, TOS = 555 Hrs.)

SLIDE # 17

	WT % of HC	
	Before Second-Stage	After Second-Stage
C ₁	6.5	6.8
C ₂ ⁻ /C ₂	1.7/2.7	1.0/2.9
C ₃ ⁻ /C ₃	7.1/1.9	2.0/5.6
C ₄ ⁻	5.9	4.7
iC ₄ /nC ₄	0/1.8	6.6/6.1
C ₅ ⁻ -C ₁₁	27.1	51.5
C ₁₂ ⁺ (Liquid)	33.3	0.8
Reactor-Wax	<u>12</u>	<u>12</u>
	100	100
C ₅ -C ₁₁ PONA, WT %:		
P	49.4	59.3
O	22.8	18.3
N	4.5	3.6
A	<u>23.3</u>	<u>18.8</u>
Total	100.0	100.0

SLIDE # 18

PROPERTIES OF RAW LIQUID HYDROCARBON
(CT 256-3-7)

TOS, Hrs 231

Properties

Specific Gravity 0.768
Aromatics, Wt % 28
Acid No. 0.15
Octane No.
R + 0 91

D-86 Distillation (°F)

IBP 97
10 162
30 223
50 262
70 300
90 363
95 424
EP 464
Residue, Wt % 3.0
Loss, Wt % -

SLIDE #19

FUTURE WORK

- The Evaluation of a Low Methane / Ethane F-T Catalyst
- A Long-Term Aging Run
- A Scoping-Type Process Economic Study