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**TECHNOLOGY DEVELOPMENT FOR IRON
FISCHER-TROPSCH CATALYSIS**

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2. Contract Objectives

The objective of this research project is to develop the technology for the production of physically robust iron-based Fischer-Tropsch catalysts that have suitable activity, selectivity and stability to be used in the slurry phase synthesis reactor development. The catalysts that are developed shall be suitable for testing in the Advanced Fuels Development Facility at LaPorte, Texas, to produce either low- or high-alpha product distributions. Previous work by the offeror has produced a catalyst formulation that is 1.5 times as active as the "standard-catalyst" developed by German workers for slurry phase synthesis. The proposed work will optimize the catalyst composition and pretreatment operation for this low-alpha catalyst. In parallel, work will be conducted to design a high-alpha iron catalyst this is suitable for slurry phase synthesis. Studies will be conducted to define the chemical phases present at various stages of the pretreatment and synthesis stages and to define the course of these changes. The oxidation/reduction cycles that are anticipated to occur in large, commercial reactors will be studied at the laboratory scale. Catalyst performance will be determined for catalysts synthesized in this program for activity, selectivity and aging characteristics.

The research is divided into four major topical areas: (a) catalyst preparation and characterization, (b) product characterization, (c) reactor operations, and (d) data assessment.

To accomplish the objectives of the project, these topics have been organized into the following technical tasks:

a. Task 1.0 Development of Optimum Promoter Levels for Low- and High-Alpha Catalysts

1.1 Determine Optimized Synthesis Procedure for High-Alpha Iron-Based Fischer-Tropsch Catalysts

- Role of precursor particle size on activity.
- Role of Cu in precipitated catalysts.
- Define attrition resistance.

1.2 Prepare Catalysts that can be Used to Determine the Role of Promoters for Low- and High-Alpha Catalysts

- Define optimum SiO_2 .
- Define optimum Al_2O_3 .

1.3 Prepare Catalysts that can be Used to Quantify the Role of K on Product Selectivity in both Low- and High-Alpha Catalysts.

1.4 Complete the Optimization of the Two Best Low-Alpha, Iron-Based Fischer-Tropsch Catalysts Developed during the Previous Contract.

b. Task 2.0 Definition of Preferred Pretreatment for both Low- and High-Alpha Fischer-Tropsch Catalysts.

2.1 Determine the Role of Cu in the Activation of Precipitated Low- and High-Alpha, Iron-Based Fischer-Tropsch Catalysts.

2.2 Determine the Effect of K Content on Activation Procedures and Determine if the Method of Addition has any Effect on Catalyst Activity and Life.

- 2.3 Determine the Physical and Chemical Changes that Occur during Catalyst Pretreatment and Use and Determine how these Changes Effect the Strength of the Catalysts.
- 2.4 Evaluate the Effect of Carbon Deposition during Catalyst Activation on Activity, Selectivity and Aging Characteristics.

c. Task 3.0 Catalyst Structure and Characterization.

d. Task 4.0 Catalyst Testing.

- 4.1 Verify the Quality of Data Obtained from the CSTR's.
- 4.2 Measure Catalyst Performance.
- 4.3 Determine the Stable Phases that Exist during Synthesis at Low and High CO Conversion Levels.
- 4.4 Obtain Data on the Rates Involved in the Interconversion of Iron Oxide and Iron Carbide.

e. Task 5.0 Reports.

3. Summary of Activities

Efforts have been made to develop a high alpha catalyst capable of operating at high conversion with $H_2:CO$ ratio of 0.7, space velocity of $3.1 \text{ NL h}^{-1} \text{ g}^{-1}(\text{Fe})$ at 1.31 MPA and 230°C . Five runs have been conducted on our best low-alpha catalyst, a precipitated iron-silicon catalyst with composition in atomic percent relative to iron: 100Fe/4.4Si/1.0K. The affects of promotion with copper and additional potassium were explored. Three catalyst compositions were studied: 100Fe/4.4Si/5.2K, 100Fe/4.4Si/2.6Cu/1.0K and 100Fe/4.4Si/2.6Cu/5.2K, given in atomic per cent relative to iron. All catalysts showed good selectivity for C_2 - C_4 alkenes ($\sim 80\%$) and low

methane yields (~2-3 wt%) indicative of a high alpha catalyst. Promising results were obtained for the 100Fe/4.4Si/2.6Cu/5.2K catalyst which had roughly twice the activity of the other two catalysts (65% CO conversion vs. 30%); however, operational problems due to plugging of the reactor wax filter prevented long term runs from being made. New methods for catalyst-wax separation are under development which will allow for better screening of catalysts when operating under heavy wax production.

4. Status, Accomplishments, Results and Discussion

A.0. - Task 1.0. Development of Optimum Promoter Levels for Low- and High-Alpha Catalysts

The goal of this task is to identify and optimize procedure for the preparation of iron-based catalysts that combine high activity selectivity and life with physical robustness. Each of the subtasks address an area of considerable uncertainty in the synthesis of catalysts.

A.1. Determine Optimized Synthesis Procedure for High-Alpha Iron-Based Catalysts

- Role of precursor size on activity.

A CSTR run using a fly ash sample having the desired particle size has been completed. The data show that this ash did not have sufficient activity to warrant further investigation.

- Role of Cu in precipitated catalysts.

See Task B.1.

- Define attrition resistance.

The inquiry regarding the spray-drying of catalysts by Niro Atomizer, Inc., Columbia, MD, has been completed. Pricing has been obtained; however, they suggest that two 55 gal. barrels of catalyst slurry would be required for them to operate their equipment. Other avenues for spray-drying are being investigated.

A.2. Prepare Catalysts that can be Used to Determine the Role of Promoters for Low- and High-Alpha Catalysts

- Define Optimum SiO_2

The catalysts described in B.1. will be used for the low- and high-alpha catalyst in this task.

- Define Optimum Al_2O_3

No scheduled or further activity to report.

A.3. Prepare Catalysts that can be Used to Quantify the Role of K on Product Selectivity for both High- and Low-Alpha Catalysts

Repeat runs in the CSTR indicate that the 3 wt.% K is at or very near the optimum K concentration for the high alpha catalyst and that is nearly 10 times the loading of K required for maximum activity for a low alpha catalyst. This implies that a single optimum alkali is not to be obtained with Fe catalysts rather, there is a strong dependence of promoter loading upon temperature.

A.4. Complete the Optimization of Two Best Low-Alpha, Iron-Based Catalysts Developed During the Previous Contract

The iron catalyst containing 0.5 wt.% K and 4.4 atomic % Si used in the aging study lasted for over 3000 hours (125 days) before losing significant activity. During

the 3000 hour period the activity decline was less than 0.8% CO conversion per week. Therefore, these data have shown that this low-alpha catalyst has the required activity and stability and that this formulation is very close to or is the optimum formulation.

A.5. Schedule of Activities for Next Quarter

- Continue efforts to examine the approaches to prepare 40 to 100 micron spherical particles.
- Continue to evaluate Fe supported on Al_2O_3 for activity.
- Determine the microhardness of the fused, reduced and carbide C-73 catalyst.

B.0. - Task 2.0. Definition of Preferred Pretreatment for Both Low- and High-Alpha Catalysts

The goals of this task are to define the preferred treatment, to define the role of Cu and K during the pretreatment on activity and selectivity and to define the chemical and physical changes which occur during the preferred pretreatment. The subtasks address each of these goals.

B.1. Role of Cu in the Activation of Precipitated Low- and High-Alpha, Iron Based Fischer-Tropsch Catalysts

The 100Fe/4.4Si/1.0K catalyst, designated RLS4.4Si150, was impregnated with aqueous $\text{Cu}(\text{NO}_3)_2 \cdot 2.5\text{H}_2\text{O}$ using an incipient wetness technique to give a final composition of 100Fe/4.4Si/2.6Cu/1.0K. A schematic of the one liter slurry reactor system is shown in Figure 1. A 10 weight percent catalyst/oil slurry was loaded into a one liter autoclave which was operated as a continuous stirred tank reactor. Additional potassium promotion was accomplished by adding the appropriate amount

of potassium t-butoxide to the catalyst/oil slurry. All catalysts were activated with synthesis gas ($H_2:CO=0.7$) for 24 h at $3.1 \text{ NL h}^{-1} \text{ g}^{-1}(\text{Fe})$, 270°C and atmospheric pressure. The pressure of the reactor was increased to 1.31 MPA and the temperature decreased to 230°C after pretreatment. The synthesis gas space velocity was maintained at $3.1 \text{ NL h}^{-1} \text{ g}^{-1}(\text{Fe})$ throughout the runs.

In previous experiments we have found that the optimum potassium loading on a catalyst promoted with 4.4 atomic percent silicon and operating at 230°C is between 4.2 and 7.0 atomic percent (Figure 2). Our best low-alpha catalyst has only 1.0 atomic percent potassium so we added an additional 4.2 weight percent to study it as a potential high-alpha catalyst (run no. LGX178). Activity results are shown in Figure 3a. The syngas conversion reached a maximum of 37% between 48 h and 144 h on stream and then decreased to 26% where it remained constant. The temperature of the reactor was increased to 270°C after 480 h on stream; however, the conversion only increased to 42% and then decreased rapidly to below 30%. The water-gas shift (WGS) activity of the catalyst was quite low while operating at 230°C . The WGS reaction quotient,

$$K_{app} = \frac{[H_2][CO_2]}{[H_2O][CO]},$$

ranged between 1.8 and 1.1 compared to the equilibrium constant of 127 at 230°C .

Alkene selectivity of the C_2 - C_4 fraction was approximately 80% throughout the run and analysis of the C_4 alkenes shows that greater than 95% was present as 1-butene.

Methane yields ranged between 2 and 3 wt.% of the total hydrocarbon. This compares to 8-11 wt% for our low-alpha catalysts when operating at high conversions.

The total methane and ethane yields were also very low varying between 2 and 4 wt%. The low methane production suggests a high α (~ 0.85); however, this can not be verified at this time because our GC which is dedicated to wax analysis is not in operation. A new column has been ordered for the GC. The maximum hydrocarbon production was 78 g m^{-3} of syngas feed. The corresponding C_3+ hydrocarbon production was 72 g m^{-3} of syngas feed or 92% of the total hydrocarbon product.

Similar activity was obtained for the 100Fe/4.4Si/2.6Cu/1.0K catalyst (run no. LGX181); however, the product selectivity differed somewhat. Syngas conversion as a function of time on stream is shown in figure 3b. Conversions were fairly constant at $\sim 30\%$ throughout the 192 h run. Water gas shift activity was very low for this run as evidenced by a low water-gas shift reaction quotient of ~ 0.7 and a relatively high $\text{H}_2:\text{CO}$ usage ratio of 1.0. The C_3 and C_4 alkene selectivities were greater than 80% as they were for the 100Fe/4.4/5.2K catalyst; however, the ethylene selectivity was much lower at about 55% to 65%. The 1-butene selectivity of the C_4 alkene fraction was greater than 90%, indicating very little isomerization. Typical hydrocarbon production during the run was 66 g m^{-3} of syngas feed with 91% present as C_3+ . Methane selectivity was slightly higher for this catalyst and ranged from 3.0 wt. % to 4.1 wt. %.

The third catalyst composition studied, 100Fe/4.4Si/2.6Cu/5.2K, gave the most promising results. Three runs were conducted with this catalyst using different catalyst-wax separation methods. Two runs used internal sintered metal filters with opening sizes of $5 \text{ }\mu\text{m}$ (run no. LGX182) and $20 \text{ }\mu\text{m}$ (run no. LGX183) while the third used an external settling tank to separate the wax and catalyst (run no. LGX184). Typically the initial syngas conversion was low at about 30% for the runs using internal

filters and 40% for the reactor fitted with the settling tank; however, conversions gradually increased during the first 72 h to approximately 60% (Figure 4). Compared to the 100Fe/4.4Si/5.2K catalyst, the addition of copper had essentially no effect on the alkene selectivity of the C₂ (78%), C₃ (75%) and C₄ (83%) fractions. Promotion with copper did increase the water-gas shift activity of the catalyst as evidenced by an increase in K_{app} to 4-12, a high CO₂ selectivity greater than 40% and a H₂:CO usage ratio less than 0.7. Despite the higher conversion, the methane selectivities were very similar to the 100Fe/4.4Si/5.2K catalyst (in the range of 2 wt% to 3 wt%). Total hydrocarbon production for these three runs ranged between 100 and 138 g m⁻³ of syngas feed with greater than 90% being C₃+

It is quite clear from these results that there is a complimentary affect between potassium and copper promoters. There is very little difference between promotion with a high level of potassium (100Fe/4.4Si/5.2K) and promotion with a low level of potassium with copper (100Fe/4.4Si/2.6Cu/1.0K); however, a significant increase in activity and productivity is seen by promotion with a high level of potassium with copper (100Fe/4.4Si/2.6Cu/5.2K). Similar results have been reported by Bukur et al. with an unsupported iron catalyst and by Soled et al. with iron-zinc catalysts.

Previously we observed no affect on activity when a 100Fe/3.6Si/0.71K catalyst was promoted with 2.6 atomic percent copper; however, this catalyst was operating at 270°C at conversions greater than 85%, so an increase in activity was not noticeable. It is apparent that when operating at relatively low temperatures in a high-alpha mode, precipitated iron catalysts need to be doubly promoted with copper and potassium. It is also apparent that there is an optimum ratio of potassium to copper as evidenced

by the difference in activity of the 100Fe/4.4Si/2.6Cu/1.0K and 100Fe/4.4Si/2.6Cu/5.2K catalysts.

B.2. Determine the Effect of K Content on Activation Procedures and
Determine if the Method of Addition has any Effect on Catalyst Activity and
Life.

No scheduled or additional activity to report.

B.3. Physical and Chemical Changes that Occur During Pretreatment and Use

The TEM data from the University of New Mexico has been received and is being evaluated. The initial evaluation of these data is that they are suggestive, but not conclusive.

B.4. Effect of Carbon Deposition

The longevity of the low-alpha catalyst (ca. 3000 hours) with an activity of $\geq$ 80% conversion suggests that carbon deposition as well as other aging factors has little impact on activity.

B.5. Schedule of Activities for Next Quarter

- Begin to define the optimum pretreatment for high-alpha catalysts with emphasis on CO + H₂ and H₂.
- Begin to define the role of promoters in high-alpha catalysts.
- Compare the effectiveness of Ca, Ba and Zn to K as promoters for low-alpha catalysts. The addition of Ca (0.5 wt.%) in addition to K had little effect on the activity of the low-alpha catalyst.

C.0. - Task 3.0. Catalyst Structure and Characterization

The goal of this task is to provide basic analyses (surface area, XRD) of all catalyst prepared and to provide additional techniques as required (Mössbauer, SEM, XPS, etc.) to answer specific questions or to provide basic required characterization data for the catalysts.

The operation of the high temperature GC has removed the necessity of developing LC methods to determine $> C_{35}$ products.

C.1. Schedule of Activities for Next Quarter

- Continue to provide the characterization data as required.

D.0. - Task 4.0. Catalyst Testing

The goals of this task are to operate the eight CSTRs, measure catalyst performance, determine the stable phases that exist during synthesis at low and high conversions and to determine the rates of interconversion of iron oxide and carbide.

D.1. Verify the Quality of Data Obtained from the CSTRs.

This task is now completed.

D.2. Measure Catalyst Performance

Major difficulties in catalyst-wax separation need to be solved before long duration runs (>300 h) can be performed with a high alpha catalyst. When either a 5 μm or a 20 μm filter was used with the 100Fe/4.4Si/2.6Cu/5.2K catalyst, plugging occurred very rapidly. This caused the slurry in the reactor to rise to a level where catalyst/wax was carried out through the gas exit; eventually this resulted in plugging of the 7 μm filter that is situated above the liquid traps (Figure 1). As a result of the filters plugging, the reactor pressure could not be controlled and the runs had to be

terminated. In order to eliminate this problem, we replaced the internal filter with an external settling tank. A schematic of the separation apparatus is shown in Figure 5. The internal filter was replaced with a piece of tubing that extended to the liquid level in the reactor. The dip tube exited through the reactor head and was connected to the midsection of a 500 cc tank. At the bottom of the tank was connected an additional tube which ran back to the reactor head. It was proposed that as the reactor level rose, a pressure differential between the reactor and the external tank would cause catalyst, wax and the gas stream to be removed continuously. It was hoped that the catalyst would settle to the bottom of the tank and return to the reactor while wax would exit through a dip tube which ran out the top of the tank through a 7 μm filter and ultimately to a 200°C trap. One run has been attempted with this separation apparatus (Run No. LGX184). In practice we found that there was no pressure differential between the separation tank and the reactor, so the entire reactor was filled before any catalyst/wax was removed. The wax that was collected from the separation tank was very dark, so it appears that the catalyst did not settle completely. This may have been caused by turbulence created by collecting the catalyst/wax continuously. An alternative method under consideration is to collect the catalyst/wax with a similar apparatus in batches. The tank could be vented to atmospheric pressure to insure catalyst/wax removal from the reactor. After removing the slurry from the reactor, the catalyst would be allowed to settle completely. Separated wax would then be removed under pressure through a dip tube and catalyst would be sent back to the reactor.

Increased efforts have been made to develop a precipitated iron catalyst for the production of high molecular weight products. Moderately high conversions were obtained for a doubly promoted 100Fe/4.4Si/2.6Cu/5.2K catalyst (65% CO conversion) operating at 230°C, 1.31 MPA with H₂:CO=0.7 and a space velocity of 3.1 NL h⁻¹ g⁻¹ (Fe). The catalyst is characterized by low methane yields and excellent light alkene selectivity. Future work on high alpha catalysts will be focused on: analysis of the liquid products to verify we are obtaining a high alpha, improving catalyst/wax separation so that long term runs can be made, improving catalyst attrition to facilitate catalyst/wax separation, optimizing the potassium and copper promoter levels and optimizing pretreatment procedures (temperature, duration, CO, H₂ or syngas, etc.).

D.3. Determine Stable Phases that Exist During Synthesis at High and Low CO

Conversion Levels

No scheduled or additional activity to report.

D.4. Obtain Data on Rates Involved in the Interconversion of Iron Oxide and

Carbide

No scheduled or additional activity to report.

D.5. Schedule of Activities for Next Quarter

- At the request of the Technical Manager, we are continuing activity testing of the Energy International's supported cobalt catalysts.

5. Publications, Presentations and Other Activities

1. Role of CO₂ in the initiation of chain growth during the Fischer-Tropsch synthesis, 14th North American Meeting of The Catalysis Society, June 11-16, 1995, Snowbird, Utah (B. H. Davis, L. Xu and S. Bao).

2. Preparation and activation of iron Fischer-Tropsch synthesis catalysts, 14th North American Meeting of The Catalysis Society, June 11-16, 1995, Snowbird, Utah (R. J. O'Brien, F. L. Tungate, R. Lin and B. H. Davis).
3. Fischer-Tropsch synthesis: Impact of potassium and zirconium promoters on the activity and structure of an ultrafine iron oxide catalyst, Topics in Catal., 2, 1 (1995) (R. J. O'Brien, L. Xu, D. R. Milburn, Y.-X. Li, K. J. Klabunde, and B. H. Davis).
4. Mössbauer spectroscopy study of iron-based catalysts used in Fischer-Tropsch synthesis, Topics in Cat., 2, 71 (1995) (K.R.P.M. Rao, F. E. Huggins, V. Mahajan, G. P. Huffman, V.U.S. Rao, B. L. Bhatt, D. B. Bukur, R. J. O'Brien and B. H. Davis).

Table 1. Activity and selectivity data for LGX178, 100Fe/4.4Si/5.2K. Operating conditions: 230 °C, 1.31 MPa, H₂:CO=0.7, 3.1 NL h⁻¹ g⁻¹(Fe).

Time on Stream/ h	24	144	288	384
CO conversion/ %	30	33	25	24
H ₂ Conversion/ %	37	42	32	30
CO+H ₂ Conversion/ %	33	37	28	26
H ₂ :CO Usage ratio	0.85	0.90	0.90	0.87
WGS/ K _{app}	1.8	1.1	1.1	1.1
CO ₂ / wt%	42	36	35	37
Total HC/ g m ⁻³ CO+H ₂ feed	64	78	60	55
C ₃ + HC/ g m ⁻³ CO+H ₂ feed	59	72	56	51
CH ₄ / wt%	3.3	2.1	2.1	2.4
CH ₄ +C ₂ H ₆ / wt%	4.1	2.8	2.7	3.8
Alkene Selectivity				
C ₂	83	84	86	72
C ₃	79	79	80	79
C ₄	84	82	82	84

Table 2. Activity and selectivity data for LGX181, 100Fe/4.4Si/2.6Cu/1.0K. Operating conditions: 230 °C, 1.31 MPa, H₂:CO=0.7, 3.1 NL h⁻¹ g⁻¹(Fe).

Time on Stream/ h	24	96	144	192
CO conversion/ %	26	27	25	22
H ₂ Conversion/ %	41	42	37	35
CO+H ₂ Conversion/ %	32	33	30	28
H ₂ :CO Usage ratio	1.1	1.1	1.0	1.1
WGS/ K _{app}	0.98	0.58	0.70	0.78
CO ₂ / mol%	38	32	32	38
Total HC/ g m ⁻³ CO+H ₂ feed	76	69	63	51
C ₃ + HC/ g m ⁻³ CO+H ₂ feed	67	62	57	45
CH ₄ / wt%	4.4	3.5	3.0	3.6
CH ₄ +C ₂ H ₆ / wt%	7.3	5.2	5.6	7.0
Alkene Selectivity				
C ₂	56	67	58	55
C ₃	82	87	81	80
C ₄	83	81	80	79

Table 3. Activity and selectivity data for LGX182, 100Fe/4.4Si/2.6Cu/5.2K. Operating conditions: 230 °C, 1.31 MPa, H₂:CO=0.7, 3.1 NL h⁻¹ g⁻¹(Fe), 5 µm filter.

Time on Stream/ h	24	96	144	168
CO conversion/ %	32	60	65	66
H ₂ Conversion/ %	32	50	54	54
CO+H ₂ Conversion/ %	32	56	61	61
H ₂ :CO Usage ratio	0.70	0.59	0.59	0.57
WGS/ K _{app}	3.8	7.9	12	10
CO ₂ / wt%	43	49	48	50
Total HC/ g m ⁻³ CO+H ₂ feed	68	112	125	122
C ₃ + HC/ g m ⁻³ CO+H ₂ feed	62	105	117	114
CH ₄ / wt%	3.0	2.4	2.2	2.3
CH ₄ +C ₂ H ₆ / wt%	4.5	3.2	2.8	3.2
Alkene Selectivity				
C ₂	68	80	79	76
C ₃	77	78	76	74
C ₄	83	82	83	85

Table 4. Activity and selectivity data for LGX183, 100Fe/4.4Si/2.6Cu/5.2K. Operating conditions: 230 °C, 1.31 MPa, H₂:CO=0.7, 3.1 NL h⁻¹ g⁻¹(Fe), 20 µm filter.

Time on Stream/ h	24	48	144	168
CO conversion/ %	32	51	63	61
H ₂ Conversion/ %	31	46	53	51
CO+H ₂ Conversion/ %	32	49	59	57
H ₂ :CO Usage ratio	0.68	0.64	0.59	0.58
WGS/ K _{app}	4.0	5.1	8.8	9.2
CO ₂ / wt%	46	46	46	46
Total HC/ g m ⁻³ CO+H ₂ feed	63	100	124	121
C ₃ + HC/ g m ⁻³ CO+H ₂ feed	57	92	116	114
CH ₄ / wt%	4.2	2.9	2.3	2.4
CH ₄ +C ₂ H ₆ / wt%	5.2	3.8	3.1	3.2
Alkene Selectivity				
C ₂	78	77	77	75
C ₃	77	74	74	75
C ₄	83	84	83	83

Table 5. Activity and selectivity data for LGX184, 100Fe/4.4Si/2.6Cu/5.2K. Operating conditions: 230 °C, 1.31 MPa, H₂:CO=0.7, 3.1 NL h⁻¹ g⁻¹(Fe), 5 µm filter.

Time on Stream/ h	24	72	144	360
CO conversion/ %	44	63	68	47
H ₂ Conversion/ %	42	56	58	43
CO+H ₂ Conversion/ %	43	60	64	46
H ₂ :CO Usage ratio	0.68	0.62	0.60	0.64
WGS/ K _{app}	4.2	6.1	8.0	4.2
CO ₂ /mol%	45	44	46	42
Total HC/ g m ⁻³ CO+H ₂ feed	89	128	134	100
C ₃ + HC/ g m ⁻³ CO+H ₂ feed	83	120	126	94
CH ₄ / wt%	2.9	2.1	2.0	2.3
CH ₄ +C ₂ H ₆ / wt%	3.7	3.3	2.6	3.2
Alkene Selectivity				
C ₂	81	68	80	78
C ₃	77	75	74	76
C ₄	83	83	83	84

Figure 1. Schematic of the one liter FTS slurry reactor system.

Figure 2. CO conversion as a function of potassium loading on a precipitated Fe-Si catalyst. $H_2/CO=0.7$, s.v.= $0.75 \text{ NL h}^{-1} \text{ g}^{-1}(\text{Fe})$, 230°C , 1.31 MPa .

Figure 3. (a) Conversion as a function of time for 100Fe/4.4Si/5.2K catalyst.
 (b) Conversion as a function of time for 100Fe/4.4Si/2.6Cu/1.0K catalyst.
 Synthesis conditions: 230°C , 1.31 MPa, $\text{H}_2:\text{CO}=0.7$, $3.1 \text{ NL h}^{-1} \text{ g}^{-1}(\text{Fe})$.

Figure 4. Conversion as a function of time for 100Fe/4.4Si/2.6Cu/5.2K catalyst. (a) 5 μm filter, (b) 20 μm filter and (c) external settling tank.

Figure 5. Schematic of the external catalyst/wax separation apparatus used for run LGX184.