# Evaluation of a Ceramic Filter in the Autoclave

A new filter assembly was used in this work. The new assembly consisted of a ceramic filter at the end of a stainless steel tube. The tube passed through the autoclave head and formed the gas and liquid exit line for the reactor. The distance between the bottom of the filter and the bottom of the autoclave was 4.6 inches. The filter nominal pore diameter was  $0.15 \mu m$ , and the absolute diameter was  $0.40 \mu m$ . The mechanical stirring of the slurry medium was achieved by an impeller at the end of a shaft that was connected to the bottom of the autoclave. The feed gas was introduced through a dip leg that extended to the bottom of the impeller.

Four runs were conducted (slurry-bed Runs 2 to 5), of which only one, slurry-bed Run 5, was successful. The Fe catalyst identification, the nature of the initial wax medium, the catalyst and initial wax weights, and the location and the pore size of the filter are summarized in Table 9. The catalyst and the C<sub>32</sub> paraffin wax medium were loaded into the 1-liter autoclave reactor at 120°C. The 1-liter autoclave was pressure tested with N<sub>2</sub> at 120°C and 18 atm. After a successful pressure test, the pressure was lowered to 11 atm, and the temperature was increased to 280°C under N<sub>2</sub> flow. Synthesis gas was introduced at 280°C. The test conditions were changed several times during this run. Autoclave internal temperatures, pressures, feed compositions, feed rates, space velocities, and stirrer speeds in revolutions per minute (rpm) at different hours on-stream are also summarized in Table 9.

The autoclave leaked during the beginning of slurry-bed Run 2. The leak problem could not be solved, and therefore, the test was terminated. Slurry-bed Runs 3 and 4 were dedicated to measuring the temperature inside the thermowell, which extended vertically through the slurry medium. These measurements verified that the height at which the temperature showed a rapid drop corresponded, in fact, to the transition between the liquid phase and the gaseous phase. The height at which the temperature drop occurred can then be used to estimate the slurry level in the autoclave.

The results in slurry-bed Run 5 are summarized in Figures 80-85. A new catalyst activation procedure was used in slurry-bed Run 5, which involved 12 hours of 2 NL/hr/gFe synthesis gas (0.7:1 H<sub>2</sub>:CO) at 280°C and 11 atm. Temperature surveys taken in the thermowell at 14, 34, and 36 hours on-stream are illustrated in Figure 80, which also shows the autoclave configuration. These results indicate that the slurry height varied between 3.5 and 5 inches during these hours. After 17 hours, the test conditions were established at 1.5 NL/hr/gFe, 257°C, and 14 atm. Under these conditions, a CO+H<sub>2</sub> conversion of about 70% was obtained with 2.8% methane selectivity and 3.1% methane plus ethane selectivity.

In slurry-bed Run 5, a pressure drop developed across the ceramic filter as a result of the buildup of solid particles. Therefore, the pressure in the exit line and in the high temperature and high pressure separator had to be gradually lowered to maintain the autoclave internal pressure at the specified level. The pressure drop gradually increased from 0.2 to 2 atm during the first 30 hours, after which the increase in the pressure drop was much more rapid. At 39

hours on-stream, the pressure drop reached a level of 17 atm, and the filter disintegrated. Significant catalyst loss occurred through the unfiltered exit line during the following hours.

In slurry-bed Run 5, the stirrer revolution per minute was originally set at 3500. The stirrer speed was lowered to 1,500 rpm after 33 hours on-stream. Subsequently, conversion dropped from 70% to about 40%. Several other revolution per minute changes were made later in the run. However, the effect of these changes on the conversion level cannot be interpreted because of the loss of catalyst from the autoclave reactor following the disintegration of the filter.

These results indicate that the ceramic filter used to drain the excess liquid from the autoclave reactor did not successfully operate. The filter rapidly plugged and disintegrated during the test.

# Evaluation of Filter with Large Pore Sizes

In an attempt to prevent plugging, filters with larger pore sizes were used in the slurry autoclave reactor. An experimental Fe catalyst was tested four times with these type of filters (slurry-bed Runs 6 to 9).

The Fe catalyst identification, the nature of initial wax medium, the catalyst and initial wax weights, the height of the static slurry, and the location and pore size of the filter are

summarized in Tables 10, 10a, and 11. The catalyst and the  $C_{32}$  paraffin wax medium were loaded into the 1-liter autoclave reactor at 120°C. The 1-liter autoclave was pressure tested with  $N_2$  at 120°C and 18 atm. After a successful pressure test, the pressure was lowered to 11 atm, and the temperature was increased to 280°C under  $N_2$  flow.

Synthesis gas with 0.7:1 H<sub>2</sub>:CO feed ratio was introduced at 280°C. During the first 12 hours, the catalyst was activated at 280°C and 11 atm using a space velocity of 1.7 to 2.0 NL/hr/gFe. Conditions were changed between 12 and 15 hours on-stream. After 15 hours, the temperature was set at 258°C, the pressure at 15 atm, and the space velocity at 1.2 to 1.4 NL/hr/gFe. Autoclave internal temperatures, pressures, feed compositions, feed rates, space velocities, and stirrer speeds in revolutions per minute at different hours on-stream are also summarized in Table 10. The catalytic performances in Runs 6 to 9 are summarized in Figures 86 to 104.

In slurry-bed Run 6, a ceramic filter, which had a nominal pore size of  $0.7~\mu m$ , was used. The bottom of the filter was 1.2 inches above the static liquid level in the autoclave. In slurry-bed Run 6 as in all other runs, the objective was to maintain the liquid level in the autoclave by continuously withdrawing the liquid products made. Liquid products were withdrawn by maintaining a pressure differential across the filter. The product gas also exited the autoclave through the filter. The outlet of the filter was connected to the high-temperature and high-pressure product collector. Following activation, a stable  $CO+H_2$  conversion of about 78% was obtained at a space velocity between 1.2 and 1.4 NL/hr/gFe. The methane and ethane

selectivity gradually increased with time on-stream and was about 4% at 72 hours. The ceramic filter plugged during the run, as was apparent from the pressure drop across the filter that gradually increased to 16 atm by 72 hours on-stream. The run was shut down at the end of 72 hours.

In slurry-bed Run 7, a metallic filter with a nominal pore diameter of 17  $\mu$ m and an absolute pore diameter of 45  $\mu$ m was used to prevent the filter from plugging with catalyst particles. The bottom of the filter was initially immersed 1/8 inch in the static slurry. The filter did not plug during the run, but the products collected through the filter contained a lot of catalyst. Following activation, the CO+H<sub>2</sub> conversion gradually decreased to about 15% by 75 hours on-stream. The decline of the conversion probably resulted from the loss of catalyst across the filter. The selectivity to methane decreased during the run, and this result suggests that the methane selectivity is lower at low conversions.

In slurry-bed Run 8, the filter had a nominal pore size of 5  $\mu$ m and an absolute pore size of 18  $\mu$ m. The bottom of the filter was 1/4 inch above the static slurry level. The filter did not plug during the run, but the products that were collected through the filter still contained a lot of catalyst. Because the initial catalyst particle size was 37 to 105  $\mu$ m, significant catalyst attrition must have occurred during the run. A gradual decline in conversion was again observed, probably because of the catalyst loss.

Slurry-bed Run 9 used the same type of filter that was used in slurry-bed Run 8, and similar results were obtained.

In summary, the 0.7- $\mu$ m ceramic filter that was used for withdrawing the liquid products from the autoclave reactor rapidly plugged with catalyst particles.

When larger pore size metallic filters (5 or 17  $\mu$ m nominal pore diameter) were used, plugging did not occur, but substantial amount of catalyst was lost. These data indicate that iron catalysts undergo significant attrition during tests in the slurry autoclave reactor.

### Evaluation of Different Filtration Procedures

In an attempt to improve the operation of the slurry reactor, several different filtration procedures there evaluated. Nine runs were conducted (slurry-bed Runs 10 to 18). The Fe catalyst identifications, the nature of initial wax medium, the catalyst and initial wax weights, the heights of the static slurry, and the locations and pore sizes of the filters are summarized in Tables 11, 11a, 11b, and 11c. The catalyst and the  $C_{32}$  paraffin initial wax medium were loaded to the 1-liter autoclave reactor at 120°C. The 1-liter autoclave was pressure tested with  $N_2$  at 120°C and 18 atm. After a successful pressure test, the pressure was lowered to 11 atm, and the temperature was increased to 280°C under  $N_2$  flow. Synthesis gas with  $H_2$ :CO feed ratio of 0.7:1 was introduced at 280°C. During the first 12 hours (except for slurry-bed Run 10, which was 6 hours), the catalyst was activated at 280°C and 11 atm using a space velocity of

1.9 to 2.0 NL/hr/gFe. Conditions were changed between 12 and 18 hours on-stream. The temperature was set at 258°C, the pressure at 15 atm, and the space velocity at 1.3 to 1.4 NL/hr/gFe. Autoclave internal temperatures, pressures, feed compositions, feed rates, space velocities, and stirrer speeds in revolutions per minute at different hours on-stream are also summarized in Table 11.

In previous tests, both the liquid product and the effluent gas exited the autoclave reactor through the filter. With slurry-bed Run 10, the autoclave was modified so that the effluent gas passed through an independent outlet port situated at the top of the autoclave. The autoclave pressure was controlled by a back-pressure regulator in the effluent gas line. The pressure drop across the filter used for draining the liquid products was controlled by independently regulating the pressure at the exit of the filter. This new filtration configuration was maintained for the rest of the work as well.

In slurry-bed Run 10, the metallic filter had a nominal pore diameter of 1  $\mu$ m and an absolute pore diameter of 5  $\mu$ m and was totally immersed in the liquid. A block valve past the filter was opened once a day, and about 0.7 atm pressure drop was exerted in an attempt to drain some of the liquid out of the autoclave. Substantial amounts of catalyst loss occurred during this procedure. Also, the filter plugged.

To prevent the catalyst loss problem, a 0.7- $\mu$ m ceramic filter was used in Run 11. The filter was back-flushed with the synthesis gas that passed through the filter. No draining was

done during the first 28 hours, and the liquid level in the autoclave increased by about 3 inches At 28 hours on-stream, the feed was diverted from the filter to the dip leg, and about 0.2 atm pressure drop was exerted. This drop in pressure caused about 2 inches of liquid to drain out (Figure 105). No further draining was done in slurry-bed Run 11, and therefore, the liquid level continued to rise during the later part of the test. A similar draining procedure was also successfully used in slurry-bed Run 12. However, in both slurry-bed Runs 11 and 12, significant problems were encountered in sealing the ceramic filter against the stainless steel tube extending from the autoclave head.

Operational problems occurred in slurry-bed Runs 13 and 14, and therefore, these runs were terminated early. Slurry-bed Runs 15 to 18 used a metallic filter to overcome the sealing problems observed with ceramic filters. The filter nominal pore size was  $0.5 \mu m$ . The filter was immersed in liquid as was the case in slurry-bed Runs 10 to 12. However, when the draining procedure that was used in slurry-bed Runs 10 to 12 with the ceramic filter was applied to the metallic filter in slurry-bed Runs 15 to 17, draining was extremely fast, causing most of the liquid inventory of the autoclave to be lost (Figure 106).

To prevent the loss of the liquid inventory, the filter level was raised to 5-5/8 inches from the bottom of the autoclave in slurry-bed Run 18 so that the bottom of the filter  $(0.5 \mu m)$  was 1-1/16 inch above the initial static slurry level. The slurry level gradually increased during the first 40 hours, after which the filter acted as a liquid level controller (Figure 107). Two filters were used. One of the filters was being continuously back-flushed with nitrogen gas, and

the other one was used for draining the liquid. At the end of a 4-hour cycle, the filters were switched. Draining of the liquid products typically required about 1 atm pressure drop. The liquid level was successfully maintained between 6 and 6.5 inches until 100 hours on-stream. Both filters plugged after 100 hours, and the run was terminated at the end of 126 hours.

In slurry-bed Run 18, a downflow propeller was placed on the stirring shaft 1-1/4 inch above the impeller to achieve good mixing. During the run, the CO conversion decreased from 80 to 60%, and the methane selectivity increased from 1.5 to 4.2% (Figures 108 and 109).

In summary, the slurry autoclave was modified so that the pressure drop across the filter can be controlled independently from the autoclave pressure. Draining of the liquid product out of the autoclave was achieved typically with a pressure drop of no more than 1 atm. Difficulties were encountered in sealing the ceramic filters against stainless steel tubes in the autoclave reactor. At comparable pore size, draining appeared to be much faster with a metallic filter. A 0.5- $\mu$ m metallic filter maintained the liquid level successfully for about 100 hours in the autoclave reactor. The liquid was maintained at a level that corresponds approximately to the bottom of the filter.

An experimental Fe catalyst showed a gradual decline in CO conversion from 80% to about 60% during the 126-hour test in Run 18. Longer testing periods are necessary to evaluate catalytic stability. During the same run, the selectivity to methane first showed a gradual

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increase and then lined out at about 4.2%. Because the selectivity to ethane is about 1%, the overall methane and ethane selectivity seems to be within target.

## Evaluation of Baseline Fe Catalyst in Run 19

A new precipitated Fe catalyst (6616-18) was prepared to evaluate its long-term performance in the slurry autoclave.

For the slurry test, 95 grams of calcined Fe catalyst were loaded into a 1-liter autoclave along with 380 grams of  $C_{32}$  *n*-paraffin wax at 130°C. After the wax melted, the autoclave was sealed and pressure tested with  $N_2$  at 25 atm. After a successful pressure test, the pressure was lowered to 14 atm, the stirring was initiated at 1,100 rpm, and the catalyst temperature was raised to 280°C in 3 hours under  $N_2$  flow. A thermocouple inserted in a thermowell inside the slurry was used for temperature control. The temperature control point was 3 cm above the bottom of the autoclave. After 280°C was achieved, the  $N_2$  feed was cut out, and synthesis gas with an  $H_2$ :CO molar ratio of 0.7:1 was introduced at 0.16 nm³/hr. After a 12-hour pretreatment, in situ, new test conditions were established, and the test continued. The conditions used in this run are summarized in Tables 11c and 11d.

Two 0.5- $\mu m$  filters (Mott Corporation, Farmington, CT) were placed at 6-5/8 inches from the bottom of the autoclave so that the liquid products could be drained periodically.

During the first 420 hours, one of the filters was continuously back-flushed with 0.2 NL/hr/gFe of  $N_2$ , and the other filter was being used for draining the liquid products.

### Performance during the First 1,250 Hours

The performance of the new precipitated Fe catalyst during the first 1,250 hours of the test is summarized in Figures 110 through 145. The figures contain the following da $^{\dagger}a$ : CO, H<sub>2</sub>, and CO+H<sub>2</sub> conversions; outlet H<sub>2</sub>:CO ratio; selectivities to methane, ethane, ethane and ethylene, ethanol, and carbon dioxide; and propylene to propane and butylene to butane ratios as well as the autoclave temperature.

After 12 hours of pretreatment with 2 NL/hr/gFe of synthesis gas at 280°C and 14 atm, the temperature was lowered to 258°C, the pressure was increased to 18 atm, and the feed rate was lowered to 1.4 NL/hr/gFe. The CO+H<sub>2</sub> conversion decreased from 78 to 72% during 12 to 27 hours on-stream. This decrease was followed by a pressure increase to 20 atm. The CO+H<sub>2</sub> conversion decreased to about 66% and remained there during 27 through 62 hours on-stream. This result appears to show an inverse relationship between activity and total pressure between 18 and 20 atm (Figure 111).

The feed rate was lowered to 1.2 NL/hr/gFe at 62 hours, and the temperature was increased to 265°C. Under these conditions, a stable CO+H<sub>2</sub> conversion of 70% was obtained between 115 and 280 hours on-stream (Figures 111 and 117). During the first 250 hours of the

run, methane selectivity gradually increased from 2 to 7% and remained constant between 250 and 280 hours on-stream (Figures 112 and 118).

The pressure increased to 21 atm at 280 hours, and the same test conditions were maintained until 420 hours on-stream. Under these conditions, a stable CO+H<sub>2</sub> conversion of 68% was obtained; selectivity to methant was about 6.1% and selectivity to ethane was 1.4% (Figure 117-119). During this period, the selectivity to ethane and ethylene, which was about 5 to 6%, could not be accurately determined because of problems associated with the gas chromatograph (GC) (Figure 120). Similarly, the selectivity to ethanol was about 2% but could not be determined more accurately because of GC problems. The propylene to propane ratio was 4 (Figure 121). The selectivity of CO to CO<sub>2</sub> was about 53%, indicating substantial water gas shift activity (Figure 116). Accordingly, the H<sub>2</sub>:CO ratio increased from 0.7 at inlet to about 1.0 at the reactor outlet.

In an attempt to increase the conversion level, the temperature was increased to 275°C, between 420 and 452 hours on-stream, and the feed rate was lowered to 0.9 NL/hr/gFe at 465 hours. Also, the N<sub>2</sub> black-flush was ended because one of the filters was plugged, leaving essentially one filter in service for the rest of the run. Under these new conditions, the catalyst maintained a stable CO+H<sub>2</sub> conversion of about 83%; selectivity to methane was about 6.9% between 452 and 1,250 hours on-stream (Figures 117-118, 123-124, 131-132, 139-140). The selectivity to ethane was 2.5 to 3.0%, the selectivity to ethane and ethylene was 5 to 6%, and the selectivity to ethanol was 2.5 to 3.0% (Figures 119-120, 125-127, 133-135, 141-143). The

olefin to paraffin ratio was 5.5 to 6.0 at a carbon number of 3 and 4.0 to 4.5 at a carbon number of 4 (Figures 121, 128-129, 136-137, 144-145). Because the selectivity to  $CO_2$  varied between 48 and 55%, the  $H_2$ :CO ratio of at the reactor outlet varied in the range of 1.2 to 1.6.

There results indicate that a new stable Fe Fischer-Tropsch catalyst was developed for slurry reactors. This catalyst did not noticeably deactivate during 1,250 hours of testing at various conditions. As a followup, the performance of this catalyst was later investigated at different conversion levels after 1,250 hours on-stream in order to predict qualitatively its performance in a slurry bubble-column reactor.

## Effect of Conversion Level on Reaction Rate

Starting from 1,260 hours on-stream, the space velocity was gradually increased, typically every 48 hours, from 0.9 to 4.0 NL/hr/gFe, by increasing the feed rate (Figures 146-152). At 1,510 hours on-stream, the temperature was lowered to 265°C, and the catalytic performance at different conversion levels was evaluated (Figures 153-160). At 1,640 hours on-stream, the space velocity was brought back to 1.2 NL/hr/gFe and maintained there until 1,740 hours on-stream. These latter test conditions were identical to those between 280 and 420 hours on-stream. Further space velocity and temperature changes were made between 1,740 and 1,950 hours on-stream (Figures 161-168). The run was shutdown at 1,996 hours because of a power outage. Catalyst performance after the shutdown is discussed in a later section (Figures 169-176).

The effect of reciprocal space velocity (NL/hr/gFe)<sup>-1</sup> on the CO+H<sub>2</sub> at 265°C and 275°C is illustrated in Figure 177. These data were later used to determine the effect of conversion level on reaction rate in Figure 178. Here, the reaction rate is expressed as the product of space velocity and CO+H<sub>2</sub> conversion. The results, which are summarized in Figure 178, indicate that the reaction rate is adversely affected by the conversion level at conversion levels greater than 50%. The decrease in reaction rate with an increase in conversion level is caused by the decrease in the partial pressure of the reactants and possibly by the increase in the partial pressure of some of the reaction products. This effect is also likely to occur at lower conversions but to a lesser degree. However, insufficient data were available at low conversions to determine the relation of reaction rate with change in conversion at low conversions.

# Catalytic Performance after Return to early Test Conditions

The CO+H<sub>2</sub> conversion between 1,640 and 1,740 hours on-stream was about 70%, which is 2% higher than that observed between 280 and 420 hours on-stream under the same test conditions (Figure 153). This result indicates that the catalyst did not deactivate during the first 1,740 hours on-stream. Between 1,640 and 1,740 hours, the selectivity to methane was about 4.5%, the selectivity to ethane was 1.5 to 2.0%, and the selectivity to ethane and ethylene was 4.5% (Figures 154-156). These selectivities were 6.1, 1.4, and 5.6% during 280 to 420 hours on-stream. These results indicate that catalytic selectivity was improving with time on-stream. Furthermore, the olefin to paraffin ratio and the water gas shift activity of the catalyst did not noticeably decrease (Figures 158-159).

### Effect of Conversion Level on Catalytic Selectivity

The selectivities to methane and to ethane and ethylene at 265°C as a function of conversion are summarized in Figure 179. Because selectivity improved with time on-stream, only relatively more recent data are illustrated in this figure. These data indicate a noticeable increase in selectivity to light ends at conversions greater than about 65%.

## Predicting Catalytic Performance in a Slurry Bubble-Column Reactor

Because the most likely candidate for a commercial-size slurry-phase reactor is a slurry bubble-column reactor, an attempt was made to predict the performance of the new precipitated Fe catalyst in a slurry bubble-column reactor based on the slurry autoclave data obtained here. The following assumptions were made:

- The slurry bubble-column reactor could be modeled as 11 slurry autoclave reactors in series operating at 8 to 80% CO+H<sub>2</sub> conversions.
- The reaction rate and selectivity did not change below 35% conversion because data at 265°C were not available at less than 35% CO+H<sub>2</sub> conversion.
- At 275°C, reaction rate and selectivity at less than 50% conversion were equal to the rate and selectivity at 50% conversion.

The calculations made using these assumptions are summarized in Table 12.

These calculations indicate that the selectivity to methane, ethane, and ethylene is estimated to be about 8.9% at  $265^{\circ}$ C and 11.8% at  $275^{\circ}$ C in a slurry bubble-column reactor. At  $265^{\circ}$ C, the selectivity to methane, ethane, and ethylene is about 2% higher than the selectivity target. At  $275^{\circ}$ C, the  $C_1+C_2$  hydrocarbon selectivity was about 5% higher than the selectivity target.

The space velocity required to reach 88% CO+H<sub>2</sub> conversion was 1.1 NL/hr/gFe at 265°C and 1.6 NL/hr/gFe at 275°C. These results appear to indicate that the new precipitated Fe catalyst may be short of the activity target by a factor of 1.8 at 265°C and by about 1.3 at 275°C. However, actual specific activities are likely to be closer to target because of unknown catalyst inventory loss across the filter during the run and also because the catalytic activities were probably underestimated at low conversions, as discussed previously.

# Catalytic Performance after Restart-Up following Cold Shutdown

The run was shut down at 1,996 hours on-stream because of a 24-hour power outage. The shutdown involved lowering the temperature to 125°C and maintaining a feed space velocity of 1.2 NL/hr/gFe. At 125°C, the synthesis gas feed was cut out, the reactor was blocked at a pressure of 21 atm, and the stirring was stopped. The feed was cooled to room temperature. Twenty-four hours later, the reactor was first heated to 90°C, stirring was initiated at 1,100 rpm, and the feed was cut in at 1.2 NL/hr/gFe to maintain a pressure of 21 atm. Then, the temperature was raised to 265°C, and the catalyst was maintained at these conditions for

another 250 hours. These test conditions were the same ones that prevailed at 280 to 420 hours and 1,640 to 1,740 hours.

The product wax drained from the reactor daily through the  $0.5~\mu m$  filter during the test period of 2,000 to 2,250 hours was initially black in color and gradually attained its typical brown color. The same phenomenon was also observed during the initial part of this run. These results indicate that the new precipitated Fe catalyst is undergoing substantial attrition following a cold startup and that some of the catalyst was lost from the reactor during these periods. Quantitation of catalyst loss in the wax products was attempted, but reliable analytical data could not be obtained. Instead, at the end of the run (after 2,250 hours), the used slurry was unloaded and analyzed for its catalyst content. The weight of catalyst recovered was 67.2 grams, which indicates that 27.8 grams of catalyst were lost during the run.

The catalytic performance after the restartup is summarized in Figures 169 to 176. A slight deactivation of about 0.5% per day was observed during this period. This conversion loss was partly caused by catalyst loss across the filter. After the restartup, methane selectivity did not increase noticeably. The H<sub>2</sub>:CO ratio at the outlet was slightly less than earlier in the run. This result may be partly caused by the lower conversion level during this period rather than a loss of water gas shift activity of the catalyst. Because of problems associated with the analysis of CO<sub>2</sub> by GC, making a definite conclusion about the status of water gas shift activity after the restartup was not possible. Similarly, fluctuations in the propylene to propane ratios were too

large to reach conclusions concerning the state of the olefin to paraffin ratios after the restartup relative to their values before shutdown.

### **Product Distribution**

The approximate product distribution based on the total amount of hydrocarbons and oxygenates recovered during 300 to 324 hours and 660 to 684 hours on-stream at 265°C and 68%  $CO+H_2$  conversion and at 275°C and 83%  $CO+H_2$  conversion, respectively, are summarized in Tables 13 and 14.

The results indicate that with increases in temperature and conversion, the selectivity to light ends and to gasoline range increases, and the selectivity to middle distillates and to wax decreases. The hydrocarbon products are 45 to 47% liquid fuels. The liquefied petroleum gas (LPG) and gasoline fractionations are olefinic. Linear  $\alpha$ -olefins make up 83% of the LPG hydrocarbons. The linear  $\alpha$ -olefin content is 43% in the gasoline-range hydrocarbons. Forty-one percent of the gasoline range has not been identified but is most likely to be internal olefins.

### Conclusions

A new precipitated Fe catalyst was developed in this program for slurry reactor operation. The new Fe catalyst is predicted to perform slightly below the performance targets for slurry bubble-column operation. Stability targets appear to be achievable. This catalyst did not noticeably

deactivate during 1,740 hours on-stream. Compared to the selectivity target, an excess of 2%  $C_1 \div C_2$  was formed at 265°C. Based on the initial catalyst inventory in the autoclave, the catalyst seems to be short of the activity target by a factor of 1.8 at 265°C and 1.3 at 275°C. However, actual specific activities are likely to be closer to target because of catalyst inventory loss across the filter during the run and because catalytic activities were underestimated at low conversions.

No work was done under Tasks 4 and 5 because the new Fe catalyst developed in this program was very stable.

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