

Table 1. List of Co-based FT Catalysts Formulated

| CAT. NO. | wt% Co | wt% M    | wt% Prom.                                      | Support   | Basis   | Prepared |
|----------|--------|----------|--|-----------|---|----------|
| Co.001   | 20%    | 1% Re    | 1% La <sub>2</sub> O <sub>3</sub> ,<br>0.13% K | γ-alumina | U.S. Pat.<br>4,880,763  | P        |
| Co.002   | 20%    | 0.43% Ru | 1% La <sub>2</sub> O <sub>3</sub>              | γ-alumina | U.S. Pat.<br>4,413,064  | P        |
| Co.003   | 20%    | 0.5% Ru  | 1% La <sub>2</sub> O <sub>3</sub>              | γ-alumina | U.S. Pat.<br>4,413,064  | P        |
| Co.004   | 20%    | 0.43% Ru | 1% La <sub>2</sub> O <sub>3</sub>              | γ-alumina | Reproduce Co.002  | P        |
| Co.005   | 20%    | 0        | 0  | γ-alumina | Base Catalyst   | P        |
| Co.005A  | 20%    | 0.4% Ru  | 0  | γ-alumina | Ru added to calcined<br>Co.005 by IW<br>impreg.                         | P        |
| Co.006   | 12%    | 0.75% Re | 0  | Titania   | U.S. Pat.<br>4,794,009  | P        |
| Co.007   | 20%    | 0        | 0  | Titania   | Base Catalyst   | P        |
| Co.008   | 20%    | 0        | 0  | Silica    | UK Pat. Appl.<br>GB 2 125 062 A<br>(Kneaded with excess<br>liquid)      | P        |
| Co.009   | 20%    | 0.5% Ru  | 0  | γ-alumina | Ru-Promoted Catalyst<br>(use Ru Chloride,<br>single-step aqueous<br>IW) | P        |
| Co.010   | 20%    | 0        | 0  | γ-alumina | Base Catalyst<br>(non-calcined)   | P        |
| Co.010A  | 20%    | 0.4% Ru  | 0  | γ-alumina | Ru added to dried<br>Co.010 by IW<br>impreg.                            | P        |
| Co.011   | 20%    | 0        | 0  | Silica    | UK Pat. Appl.<br>GB 2 125 062 A<br>(Kneaded)                            | P        |
| Co.012   | 20%    | 0        | 0  | Silica    | Base Catalyst<br>(Inc. Wetness)   | P        |
| Co.013   | 20%    | 0        | 0  | Titania   | Base Catalyst<br>like Co.007, but all<br>aqueous                        | P        |
| Co.014   | 12%    | 0.5% Ru  | 0  | Titania   | Ru-Promoted Catalyst<br>(aqueous IW co-<br>impregnation)                | P        |
| Co.015   | 20%    | 0.43% Ru | 1% La <sub>2</sub> O <sub>3</sub>              | γ-Alumina | Similar to Co.002,<br>but all aqueous                                   | P        |
| Co.016   | 20%    | 0.43% Ru | 1% La <sub>2</sub> O <sub>3</sub>              | γ-Alumina | Similar to Co.015,<br>but calcined after Co<br>impregnation             | P        |

Table 1. List of Co-based FT Catalysts Formulated (Contd.)

| CAT. NO. | wt% Co | wt% M   | wt% Prom.                         | Support                   | Basis  | Prepared |
|----------|--------|---------|-----------------------------------|---------------------------|--|----------|
| Co.017   | 20%    | 0.5% Ru | 1% La <sub>2</sub> O <sub>3</sub> | γ-Alumina                 | Similar to Co.003, but all aqueous                             | P        |
| Co.018   | 20%    | 0.5% Ru | 0                                 | γ-Alumina                 | Ru-Promoted Catalyst (single-step, aqueous IW)                 | P        |
| Co.019   | 20%    | 0.5% Ru | 0                                 | Silica                    | Ru-Promoted Catalyst (single-step, aqueous IW)                 | P        |
| Co.020   | 0      | 0.5% Ru | 0                                 | γ-Alumina                 | Ru Base Catalyst   | P        |
| Co.020A  | 20%    | 0.4% Ru | 0                                 | γ-Alumina                 | Co added to dried Co.020 by IW impreg.                         | P        |
| Co.020B  | 20%    | 0.4% Ru | 0                                 | γ-Alumina                 | Co added to reduced Co.020 by IW impreg.                       | P        |
| Co.020C  | 20%    | 0.4% Ru | 0                                 | γ-Alumina                 | Co added to calcined Co.020 by IW impreg.                      | P        |
| Co.021   | 20%    | 0       | 0.7% Zr                           | Silica                    | U.K. Pat. Appl. GB 2 125 062 A (single-step)                   | P        |
| Co.022   | 20%    | 0       | 0.7% Zr                           | Silica                    | U.K. Pat. Appl. GB 2 125 062 A (multiple-steps)                | P        |
| Co.023   | 20%    | 0       | 1.4% Zr                           | Silica                    | U.K. Pat. Appl. GB 2 125 062 A (multiple-steps)                | P        |
| Co.024   | 20%    | 0       | 8.5% Zr                           | Silica                    | Eur. Pat. Appl. 0 167 215 A2 (non-aqueous Zr pre-impregnation) | P        |
| Co.025   | 20%    | 0       | 8.5% Zr                           | Silica                    | Similar to Co.024 but aqueous Zr pre-impregnation              | P        |
| Co.026   | 20%    | 0       | 0                                 | Silica (Davison Grade 59) | Similar to Co.012 but different grade silica                   | P        |
| Co.027   | 20%    | 0.5% Ru | 0                                 | Silica (Davison Grade 59) | Similar to Co.019 but different grade silica                   | P        |
| Co.028   | 20%    | 0.5% Ru | 0.5% K                            | γ-alumina                 | Ru- and K-Promoted Catalyst (single-step IW)                   | P        |
| Co.029   | 30%    | 0.5% Ru | 0.5% K                            | γ-alumina                 | Similar to Co.028 but 30% Co                                   | P        |

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| CAT. NO. | wt% Co | wt% M    | wt% Prom.           | Support           | Basis   | Prepared |
|----------|--------|----------|---------------------|-------------------|---|----------|
| Co.030   | 20%    | 0        | 0.1% Zr             | $\gamma$ -alumina | Zr-Promoted Catalyst (single-step aqueous IW)                         | P        |
| Co.031   | 20%    | 0        | 1.4% Zr             | $\gamma$ -alumina | Zr-Promoted Catalyst (single-step aqueous IW)                         | P        |
| Co.032   | 20%    | 0        | 8.5% Zr             | $\gamma$ -alumina | Zr-Promoted Catalyst (aqueous IW co-impregnation)                     | P        |
| Co.033   | 20%    | 0        | 8.5% Zr             | $\gamma$ -alumina | multiple-steps, aqueous IW, Co pre-impregnation                       | P        |
| Co.034   | 20%    | 0        | 8.5% Zr             | $\gamma$ -alumina | multiple-steps, aqueous IW, Zr pre-impregnation                       | P        |
| Co.035   | 20%    | 0        | 8.5% Zr             | Silica            | Zr-Promoted Catalyst (single-step aqueous, kneaded)                   | P        |
| Co.036   | 20%    | 0        | 8.5% Zr             | Silica            | multiple-steps, aq. Co pre-impreg. by kneading, aqueous IW Zr impreg. | P        |
| Co.037   | 12%    | 0        | 0                   | Titania           | Similar to Co.013, but TiO <sub>2</sub> mainly anatase                | P        |
| Co.038   | 12%    | 0.5% Ru  | 0                   | Titania           | Similar to Co.014, but TiO <sub>2</sub> mainly anatase                | P        |
| Co.039   | 12%    | 0        | 0                   | Titania           | Similar to Co.007, but only 12% Co                                    | P        |
| Co.040   | 12%    | 0        | 0                   | Titania           | Similar to Co.013, but only 12% Co                                    | P        |
| Co.041   | 20%    | 0.5% Ru  | 8.5% Zr             | Silica            | Similar to Co.025, but Ru-promoted                                    | P        |
| Co.042   | 0      | 0        | 8.5% Zr             | Silica            | Blank Zr-promoted SiO <sub>2</sub>                                    | P        |
| Co.043   | 20%    | 0.5% Ru  | 8.5% Zr<br>0.5% K   | Silica            | Similar to Co.041, but K-promoted                                     | P        |
| Co.044   | 20%    | 0        | 15% Zr              | Silica            | Similar to Co.025, but 15% Zr   | P        |
| Co.045   | 20%    | 0        | 4% Zr               | Silica            | Similar to Co.025, but 4% Zr  | P        |
| Co.046   | 12%    | 0.75% Re | 4% SiO <sub>2</sub> | Titania           | Similar to Co.006, but SiO <sub>2</sub> -promoted                     | -        |

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| CAT. NO.            | wt% Co | wt% M   | wt% Prom.                           | Support           | Basis  | Prepared |
|---------------------|--------|---------|-------------------------------------|-------------------|--|----------|
| Co.047              | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.028, but 0.3% K  | P        |
| Co.048              | 20%    | 0.5% Ru | 8.5% Zr<br>0.3% K                   | Silica            | Similar to Co.043, but 0.3% K  | P        |
| Co.049              | 20%    | 0.5% Ru | 0.1% K                              | $\gamma$ -alumina | Similar to Co.047, but 0.1% K  | P        |
| Co.050              | 20%    | 0.5% Ru | 8.5% Zr<br>0.1% K                   | Silica            | Similar to Co.048, but 0.1% K  | P        |
| Co.051              | 0      | 0.5% Ru | 0                                   | Titania (R)       |  | P        |
| Co.052              | 0      | 0.5% Ru | 0                                   | Titania (A)       |  | P        |
| Co.053              | 20%    | 0.5% Ru | 0                                   | $\gamma$ -alumina | Similar to Co.018 (New Batch)  | P        |
| Co.054              | 20%    | 0       | 8.5% Zr<br>0.3% K                   | Silica            | Similar to Co.043, but without Ru  | P        |
| Co.055              | 20%    | 1% Re   | 1% La <sub>2</sub> O <sub>3</sub>   | $\gamma$ -alumina | Similar to Co.001, but without K   | P        |
| Co.056              | 20%    | 0       | 8.5% La <sub>2</sub> O <sub>3</sub> | Silica            | Similar to Co.025, but La instead of Zr                                    | P        |
| CAL.01<br>(97E-13E) | 20%    | 0.5% Ru | 0                                   | $\gamma$ -alumina | Similar to Co.018 (Calsicat Prep.)   | P        |
| CAL.02<br>(97E-16B) | 20%    | 0.5% Ru | 0                                   | $\gamma$ -alumina | Similar to CAL.01 (New Batch)  | P        |
| CAL.03<br>(97E-13F) | 20%    | 0.5% Ru | 0                                   | $\gamma$ -alumina | 2-step impreg., Similar to CAL.02  | P        |
| CAL.04<br>(97E-50C) | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.047 Stand. Calc. in Air                                      | P        |
| CAL.05<br>(97E-50D) | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.047 Stand. Calc. in N <sub>2</sub>                           | P        |
| CAL.06<br>(97E-51A) | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.047 Red. and Pas. in Air                                     | P        |
| CAL.07<br>(97E-51B) | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.047 Red. and Protected in Paraffin (51.3% Cat. Conc.)        | P        |
| CAL.08<br>(97E-51C) | 20%    | 0.5% Ru | 0.3% K                              | $\gamma$ -alumina | Similar to Co.047 Calc., Red. and Protected in Paraffin (54.4% Cat. Conc.) | P        |

Table 2. List of Water-Gas Shift Catalysts and F-T Catalysts with WGS Function

| CAT. NO. | wt% Co | wt% M   | wt% Prom. | Support           | Basis   | Prepared |
|----------|--------|---------|-----------|-------------------|---|----------|
| WGS.01   | 0      | 5.0% Cu | 0         | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.02   | 0      | 5.0% Cu | 10% Zn    | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.03   | 0      | 5.0% Cu | 4.0% Cr   | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.04   | 0      | 0       | 4.0% Cr   | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.05   | 0      | 0       | 4.0% Mn   | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.06   | 0      | 5.0% Cu | 4.0% Mn   | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.07   | 0      | 4.0% Fe | 0         | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.08   | 0      | 4.0% Fe | 4.0% Cr   | $\gamma$ -alumina | single-step, aqu. IW                                  | P        |
| WGS.09   | 0      | 5.0% Cu | 4.0% Cr   | $\gamma$ -alumina | Similar to WGS.03                                     | -        |
| CoW.01   | 20%    | 5.0% Cu | 10% Zn    | $\gamma$ -alumina | multiple-steps, aqu. IW, Cu,Zn pre-impr.              | P        |
| CoW.02   | 20%    | 5.0% Cu | 4.0% Cr   | $\gamma$ -alumina | multiple-steps, aqu. IW, Cu,Cr pre-impr.              | P        |
| CoW.03   | 10%    | 5.0% Cu | 4.0% Cr   | $\gamma$ -alumina | similar to CoW.02 (10% Co only)                       | P        |
| CoW.04   | 10%    | 10% Cu  | 8.0% Cr   | $\gamma$ -alumina | similar to CoW.03; 10% Cu and 8% Cr                   | P        |
| CoW.05   | 20%    | 5.0% Cu | 4.0% Cr   | $\gamma$ -alumina | similar to CoW.02 with support calcined at 750°C      | P        |
| CoW.06   | 20%    | 5.0% Cu | 4.0% Cr   | Silica            | multiple-steps, aqu. IW, Cu,Cr pre-impr.              | P        |
| CoW.07   | 20%    | 5.0% Cu | 4.0% Cr   | Silica            | similar to CoW.06, but with support calcined at 750°C | P        |

Table 3. Summary of Physical Properties

| CATALYST                               | Composition   | BET Surface Area (m <sup>2</sup> /g) | Pore Volume (cc/g) | Average Pore Dia. (Å) | Average Part. Size (μm) |
|--|---|--------------------------------------|--------------------|-----------------------|-------------------------|
| Vista-B Al <sub>2</sub> O <sub>3</sub> | (0-400 mesh)  | 299                                  | 1.69               | 269                   | 24                      |
| Vista-B Al <sub>2</sub> O <sub>3</sub> | Calcined 500°C  | 240                                  | 0.49               | 82                    |                         |
| Vista-B Al <sub>2</sub> O <sub>3</sub> | Calcined 600°C  | 206                                  | 0.49               | 95                    |                         |
| Vista-B Al <sub>2</sub> O <sub>3</sub> | Calcined 600°C  | 174                                  | 0.47               | 109                   |                         |
| Davison SiO <sub>2</sub>               | (0-400 mesh)  | 219                                  | 0.51               | 89                    | 121                     |
| Degussa P25 TiO <sub>2</sub>           | (as received)   | 47                                   | 0.40               | 335                   | 6                       |
| Degussa P25 TiO <sub>2</sub>           | Dried 60°C, Calc. 350°C/16hrs                         | 46                                   | 0.48               | 419                   | 51                      |
| Degussa P25 TiO <sub>2</sub>           | Dried 60°C, Calc. 650°C/16hrs                         | 12                                   | 0.17               | 573                   | 212                     |
| Co.001                                 | 20Co/1Re/<br>1La <sub>2</sub> O <sub>3</sub> /0.13K/A | 191                                  | 0.31               | 66                    | 42<br>40                |
| Co.002                                 | 20Co/0.43Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A    | 149                                  | 0.33               | 89                    | 74                      |
| Co.003                                 | 20Co/0.5Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A     | 122                                  | 0.26               | 87                    | 77                      |
| Co.004                                 | 20Co/0.43Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A    |                                      |                    |                       |                         |
| Co.005                                 | 20Co/A  | 173                                  | 0.34               | 81                    | 65<br>86                |
| Co.006                                 | 12Co/0.75Re/T   | 16                                   | -                  | -                     |                         |
| Co.007                                 | 20Co/T  | 11                                   | 0.10               | 372                   |                         |
| Co.008                                 | 20Co/S  | 181                                  | 1.06               | 234                   |                         |
| Co.009                                 | 20Co/0.5/A  |                                      |                    |                       |                         |
| Co.010                                 | 20Co/A  |                                      |                    |                       |                         |
| Co.011                                 | 20Co/S  | 211                                  | 1.07               | 203                   | 107                     |
| Co.012                                 | 20Co/S  | 211                                  | -                  | -                     | 105                     |
| Co.013                                 | 20Co/T  |                                      |                    |                       |                         |
| Co.014                                 | 12Co/0.5Ru/T  | 15                                   | 0.12               | 328                   |                         |
| Co.015                                 | 20Co/0.43Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A    | 141                                  | -                  | -                     | 67                      |

Table 3. Summary of Physical Properties (contd.)

| CATALYST | Composition   | BET Surface Area (m <sup>2</sup> /g) | Pore Volume (cc/g) | Average Pore Dia. (Å) | Average Part. Size (μm) |
|----------|---|--------------------------------------|--------------------|-----------------------|-------------------------|
| Co.016   | 20CoB/0.43Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A | 116                                  | -                  | -                     | 79                      |
| Co.017   | 20Co/0.5Ru/<br>1La <sub>2</sub> O <sub>3</sub> /A   | 123                                  | -                  | -                     | 73                      |
| Co.018   | 20Co/0.5Ru/A  | 158                                  | -                  | -                     | 69                      |
| Co.019   | 20Co/0.5Ru/S  |                                      |                    |                       | 110                     |
| Co.021   | 20Co/0.7Zr/S  | 213                                  | 1.12               | 210                   |                         |
| Co.023   | 20Co/1.4Zr/S  | 213                                  | -                  | -                     |                         |
| Co.024   | 20Co/8.5Zr/S  | 215                                  | 1.08               | 202                   |                         |
| Co.025   | 20Co/8.5Zr/S  | 208                                  | 0.97               | 187                   |                         |
| Co.026   | 20Co/S(59)  | 184                                  | -                  | -                     |                         |
| Co.027   | 20Co/S(59)  | 189                                  | -                  | -                     |                         |
| Co.028   | 20Co/0.5Ru/0.5K/A                                   | 161                                  | 0.33               | 82                    |                         |
| Co.029   | 30Co/0.5Ru/0.5K/A                                   | 140                                  | -                  | -                     |                         |
| Co.032   | 20Co/8.5Zr/A  | 155                                  | 0.30               | 77                    |                         |
| Co.034   | 20Co/8.5Zr/A  | 150                                  | 0.31               | 83                    |                         |
| Co.035   | 20Co/8.5Zr/S  | 207                                  | 1.21               | 233                   |                         |
| Co.036   | 20Co/8.5Zr/S  | 209                                  | -                  | -                     |                         |
| Co.037   | 12Co/T(A)   | 38                                   | -                  | -                     |                         |
| Co.039   | 12Co/T(R)   | 13                                   | -                  | -                     |                         |
| Co.041   | 20Co/0.5Ru/8.5Zr/S                                  | 214                                  | -                  | -                     | -                       |
| Co.047   | 20Co/0.5Ru/0.3K/A                                   | 162                                  | 0.31-              | 78                    |                         |
| CAL.01   | 20Co/0.5Ru/A  | 178                                  | 0.34               | 76                    |                         |
| CAL.02   | 20Co/0.5Ru/A  | 158                                  | -                  | -                     |                         |
| CAL.03   | 20Co/0.5Ru/A  | 158                                  | -                  | -                     |                         |
| CAL.04   | 20Co/0.5Ru/0.3K/A                                   | 151                                  |                    |                       |                         |
| CAL.05   | 20Co/0.5Ru/0.3K/A                                   | 163                                  |                    |                       |                         |
| CAL.06   | 20Co/0.5Ru/0.3K/A                                   | 162                                  |                    |                       |                         |
| CAL.07   | 20Co/0.5Ru/0.3K/A                                   | -                                    |                    |                       |                         |

Table 3. Summary of Physical Properties (contd.)

| CATALYST | Composition       | BET Surface Area (m <sup>2</sup> /g) | Pore Volume (cc/g) | Average Pore Dia. (Å) | Average Part. Size (μm) |
|----------|-------------------|--------------------------------------|--------------------|-----------------------|-------------------------|
| CAL.08   | 20Co/0.5Ru/0.3K/A | -                                    |                    |                       |                         |



Table 4. H<sub>2</sub> Chemisorption and TPR Results

| Catalyst                   | H <sub>2</sub> Chemisorption <sup>a</sup>      |  |                            |            | H <sub>2</sub> TPR                           |   |
|----------------------------|--|--|----------------------------|------------|--|---|
|                            | Total<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Irrevers.<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Average<br>$d_p^b$<br>(nm) | %<br>Disp. | %Co Red. <sup>c</sup><br>(TPR calc.<br>Cat.) | % Red. <sup>d</sup><br>(stand.<br>red.) |
| Co.001                     | 174 $\pm$ 5                                    | 157 $\pm$ 5  |                            | 10.2       |  |   |
| Co.002                     | 155  | 130  |                            | 9.1        |  |   |
| Co.003                     | 165  | 140  |                            | 9.6        |  |   |
| Co.004 <sup>1</sup><br>" 2 | 205<br>65                                      | 185<br>56  |                            | 12.1<br>4  |  |   |
| Co.005                     | 48   | 42   | 20                         | 2.8        | 89   | 58                                      |
| Co.005a                    | 129  | 110  | 11                         | 7.6        | 92   | 84                                      |
| Co.006                     | 44   | 32   | 19                         | 4          | 80   |   |
| Co.007                     | 33   | 23   | 36                         | 2.0        | 78   | 78                                      |
| Co.008                     |  |  |                            |            |  |   |
| Co.009                     | 133  | 109  | 13                         | 7.8        | 98   |   |
| Co.010                     |  |  |                            |            |  |   |
| Co.010a                    | 124  | 100  | 12.5                       | 7.3        | 91   | 89                                      |
| Co.011                     | 82   | 70   | 15.6                       | 4.8        | 75   | 75                                      |
| Co.012                     | 89   | 74   | 15                         | 5.2        | 80   | 80                                      |
| Co.013                     | 21   | 17   | 79                         | 1.2        | 97   |   |
| Co.014                     | 38   | 30   | 21                         | 3.7        | 79   |   |
| Co.015                     | 146  | 124  | 11.7                       | 8.6        | 94   | 94                                      |
| Co.016                     | 163  | 146  | 10.5                       | 9.6        | 96   | 96                                      |
| Co.017                     | 202  | 183  | 8.5                        | 11.9       | 97   | 97                                      |
| Co.018                     | 185  | 165  | 9.2                        | 11         | 97   | 94                                      |

Table 4. H<sub>2</sub> Chemisorption<sup>a</sup> and TPR Results (contd.)

| Catalyst | H <sub>2</sub> Chemisorption <sup>a</sup>      |   |  |            | TPR  |   |
|----------|--|---|--|------------|--|---|
|          | Total<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Irrev.<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Average<br>d <sub>p</sub> <sup>b</sup><br>(nm) | %<br>Disp. | %Co Red. <sup>c</sup><br>(TPR calc.<br>Cat.) | % Red. <sup>d</sup><br>(stand.<br>red.) |
| Co.018Cl |  |   |  |            | 95   |   |
| Co.019   | 112  | 92  |  | 6.6        |  |   |
| Co.020   |  |   |  |            |  |   |
| Co.020a  | 153  | 126   | 10   | 9.1        | 88   |   |
| Co.020b  | 134  | 113   | 11   | 7.9        | 86   |   |
| Co.020c  | 115  | 100   | 13   | 6.8        | 88   |   |
| Co.021   | 74   | 50  |  | 4.3        |  |   |
| Co.022   | 141  | 122   |  | 8.3        | 80   |   |
| Co.023   | 158  | 136   |  | 9.3        | 81   |   |
| Co.024   | 87   | 72  |  | 5.1        | 91   |   |
| Co.025   | 93   | 77  |  | 5.5        | 75   |   |
| Co.026   |  |   |  |            |  |   |
| Co.027   |  |   |  |            |  |   |
| Co.028   | 170  | 148   |  | 10         |  |   |
| Co.029   | 175  | 155   |  | 7          |  |   |
| Co.030   |  |   |  |            |  |   |
| Co.031   | 71   | 51  | 20   | 4.2        | 82   |   |
| Co.032   | 55   | 39  | 26   | 3.2        | 85   |   |
| Co.033   | 43   | 32  | 31   | 2.5        | 79   |   |
| Co.034   | 114  | 91  | 14   | 6.7        | 96   |   |
| Co.035   | 125  | 115   | 11   | 7.2        | 82   |   |
| Co.036   | 122  | 98  |  | 7.2        |  |   |

Table 4. H<sub>2</sub> Chemisorption<sup>a</sup> and TPR Results (contd.)

| Catalyst | H <sub>2</sub> Chemisorption <sup>a</sup>      |   |                            |                  | TPR  |   |
|----------|--|---|----------------------------|------------------|--|---|
|          | Total<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Irrev.<br>( $\mu$ mol<br>H <sub>2</sub> /g cat) | Average<br>$d_p^b$<br>(nm) | %<br>Disp.       | %Co Red. <sup>c</sup><br>(TPR calc.<br>Cat.) | % Red. <sup>d</sup><br>(stand.<br>red.) |
| Co.037   | 21   | 3   | 48                         | 2                | 99   |   |
| Co.038   | 45   | 35  | 21                         | 4.3              | 91   |   |
| Co.039   | 19   | 16  | 40                         | 1.8              | 72   |   |
| Co.040   | 14   | 11  |                            | 1.4              |  |   |
| Co.041   | 70   | 55  |                            | 4                |  |   |
| Co.042   |  |   |                            |                  |  |   |
| Co.043   | 137  | 116   |                            | 8                |  |   |
| Co.053   |  |   |                            |                  |  |   |
| CAL.01   | -  | -   | 58 <sup>e</sup>            | 4.4 <sup>e</sup> |  |   |
| CAL.02   | -  | -   | 63 <sup>e</sup>            | 5.1 <sup>e</sup> |  |   |
| CAL.03   | -  | -   | 57 <sup>e</sup>            | 4.2 <sup>e</sup> |  |   |
| CAL.04   |  |   | 55 <sup>e</sup>            |                  |  |   |
| CAL.05   |  |   | 56 <sup>e</sup>            |                  |  |   |
| CAL.06   |  |   | 57 <sup>e</sup>            |                  |  |   |
| CAL.07   |  |   | -                          |                  |  |   |
| CAL.08   |  |   | -                          |                  |  |   |

(a) static H<sub>2</sub> chemisorption at 100°C

(b) Est. assuming  $H_{10}/Co_s = 1$ ,  $5.46 \times 10^{-20} \text{ m}^2/Co_s$ , and  $d_p = 5/S_{Co}/\rho$

$S_{Co}$  is based on amt. Co reducible during standard reduction

(c) % Co reducible from TPR of calcined catalysts up to 900°C

(d) % Co reduced after standard reduction procedure

(e) Based on CO chemisorption

(f) Catalyst Co.018-Cl is chlorinated.

Table 5. Summary of Fixed Bed Reaction Results

| Catalyst | Run | Prep   | CO Conv (%) | Rate g CH <sub>2</sub> /g cat/hr | R:CO <sub>2</sub> g CH <sub>2</sub> /g cat/hr | R:(CO) mol/mol Co/s | wt% CH <sub>4</sub> | alpha | C <sub>2</sub> -C <sub>5</sub> Olef/Par | CO <sub>2</sub> (%) | B/g cat/hr | E <sub>act</sub> kcal/mol | Comment                                |
|----------|-----|--------|-------------|----------------------------------|---|---------------------|---------------------|-------|---|---------------------|------------|---------------------------|--|
| Co.001   | 4   | (1)    | 2.6         | 0.116                            | 0.056   | 6.8E-04             | 19.6                | 0.76  | 8.61                                    | 0.4                 | 0.061      |                           | NSH**                                  |
| Co.002   | 4   | (2)    | 6.7         | 0.258                            | 0.082   | 1.5E-03             | 26.1                | 0.64  | 1.97                                    | 0.1                 | 0.012      |                           | NS*                                    |
| Co.003   | 1   | (3)    | 5.5         | 0.185                            | 0.044   | 1.1E-03             | 30.8                | 0.57  | 4.22                                    |                     |            | 28.9                      | old startup                            |
| Co.004   | 1   | (2) s  | 4.3         | 0.291                            | 0.086   | 1.7E-03             | 30.4                | 0.62  | 2.77                                    | 0.1                 | 0.025      | 25.9                      | NSH                                    |
|          | 2   | u      | 11.2        | 0.517                            | 0.155   | 3.0E-03             | 28.9                | 0.61  | 1.02                                    | 0.1                 | 0.038      |                           | NSH                                    |
|          | 2h  | u      | 47.6        | 0.548                            | 0.211   | 3.2E-03             | 21.9                | 0.64  | 0.40                                    | 2.3                 | 0.090      |                           | NSH, high conv. study                  |
|          | 2a  | f      | 3.8         | 0.177                            | 0.063   | 1.0E-03             | 25.5                | 0.65  | 3.02                                    | 0.1                 | 0.024      |                           | NSH, calc. after run2, T surge         |
|          | 3   | u      | 6.7         | 0.499                            | 0.153   | 2.9E-03             | 27.8                | 0.61  | 1.45                                    | 0.1                 | 0.036      |                           | NSH                                    |
|          | 3h  | u      | 27.0        | 0.506                            | 0.164   | 3.0E-03             | 27.3                | 0.62  | 0.46                                    | 0.8                 | 0.048      |                           | NSH, high conv. study                  |
|          | 4   | u      | 1.8         | 0.427                            | 0.241   | 2.5E-03             | 24.0                | 0.79  | 2.40                                    | 0.1                 | 0.078      |                           | NSH, 10 atm                            |
|          | 5   | f      | 4.5         | 0.288                            | 0.150   | 1.7E-03             | 19.4                | 0.75  | 7.84                                    | 0.1                 | 0.027      |                           | NSH                                    |
| Co.005   | 2   | (1)    | 3.3         | 0.087                            | 0.037   | 5.1E-04             | 21.4                | 0.66  | 3.40                                    | 0.1                 | 0.005      |                           | NS                                     |
| "        | 2a  |        | 2.5         | 0.066                            | 0.021   | 3.9E-04             | 25.9                | 0.59  | 4.92                                    | 0.1                 | 0.004      |                           | CO:H <sub>2</sub> :Ar = 1:2:2          |
| "        | 2b  |        | 2.4         | 0.064                            | 0.021   | 3.7E-04             | 26.3                | 0.60  | 6.22                                    | 0.1                 | 0.006      |                           | + H <sub>2</sub> O (eq. 7.5% CO conv.) |
| "        | 3   |        | 3.3         | 0.077                            | 0.024   | 4.5E-04             | 28.4                | 0.62  | 2.29                                    | 0.1                 | 0.013      |                           | NSH                                    |
| "        | 4   |        | 4.0         | 0.069                            | 0.021   | 4.0E-04             | 30.0                | 0.62  | 2.16                                    | 0.2                 | 0.009      | 25.1                      | NSH                                    |
| "        | 5   |        | 3.9         | 0.171                            | 0.084   | 1.0E-03             | 26.8                | 0.76  | 2.59                                    | 0.1                 | 0.018      |                           | NSH, 10 atm                            |
|          | 6   |        | 2.1         | 0.129                            | 0.044   | 7.6E-04             | 25.7                | 0.64  | 5.53                                    | 0.1                 | 0.017      |                           | NSH                                    |
|          | 7   |        | 6.7         | 0.084                            | 0.040   | 4.9E-04             | 18.8                | 0.70  | 1.95                                    | 0.1                 | 0.003      |                           | S2                                     |
| Co.005a  | 1   | (4a)   | 3.2         | 0.227                            | 0.067   | 1.3E-03             | 30.0                | 0.60  | 3.05                                    | 0.1                 | 0.034      |                           | NSH                                    |
| Co.005b  | 1   | (1)    | 2.4         | 0.040                            | 0.024   | 2.3E-04             | 13.9                | 0.74  | 0.55                                    | 0.4                 | 0.023      |                           | S2, 1/4WGS.03 + 3/4Co.005              |
| Co.005c  | 1   | (1)    | 5.7         | 0.071                            | 0.031   | 4.1E-04             | 20.3                | 0.68  | 0.21                                    | 1.8                 | 0.076      |                           | S2, 1/2WGS.03 + 1/2Co.005              |
| Co.006   | 1   | (5)    | 2.7         | 0.052                            | 0.007   | 5.1E-04             | 45.0                | 0.49  | 1.90                                    | 0.1                 | 0.006      |                           | NSH                                    |
| Co.007   | 1   | (5)    | 3.2         | 0.024                            | 0.003   | 1.4E-04             | 49.8                | 0.50  | 0.72                                    | 0.2                 | 0.004      |                           | NSH                                    |
| Co.009   | 1   | (1)    | 4.9         | 0.346                            | 0.090   | 2.0E-03             | 31.1                | 0.57  | 1.69                                    | 0.1                 | 0.020      |                           | NSH                                    |
| "        | 2   |        | 4.9         | 0.338                            | 0.091   | 2.0E-03             | 31.1                | 0.58  | 1.64                                    | 0.1                 | 0.026      |                           | NSH                                    |
| Co.010a  | 1   | (4b) s | 3.5         | 0.246                            | 0.076   | 1.4E-03             | 29.5                | 0.62  | 2.58                                    | 0.2                 | 0.040      |                           | NSH                                    |
| "        | 2   |        | 4.1         | 0.262                            | 0.080   | 1.5E-03             | 30.0                | 0.61  | 2.31                                    | 0.1                 | 0.022      |                           | NSH                                    |
| Co.011   | 2   | (6b)   | 2.9         | 0.094                            | 0.037   | 5.5E-04             | 22.4                | 0.61  | 4.83                                    | 0.3                 | 0.030      |                           | slow CO startup (60 min)               |
| Co.012   | 1   |        | 3.8         | 0.083                            | 0.027   | 4.8E-04             | 26.3                | 0.64  | 4.40                                    |                     |            | 22.5                      | slow CO startup (60 min)               |
| "        | 2   |        | 4.4         | 0.085                            | 0.030   | 5.0E-04             | 29.1                | 0.64  | 1.93                                    | 0.3                 | 0.018      |                           | NSH                                    |
| "        | 3   |        | 2.1         | 0.081                            | 0.026   | 4.7E-04             | 28.6                | 0.66  | 3.57                                    | 0.4                 | 0.049      |                           | NSH                                    |
|          | 4   |        | 2.5         | 0.105                            | 0.035   | 6.2E-04             | 28.4                | 0.61  | 4.94                                    | 0.4                 | 0.023      |                           | NSH                                    |
| Co.014   | 1   | (1)    | 1.5         | 0.028                            | 0.009   | 2.8E-04             | 32.3                | 0.63  | 4.70                                    | 0.1                 | 0.003      |                           | NSH                                    |
| "        | 2   |        | 2.6         | 0.034                            | 0.013   | 3.3E-04             | 27.7                | 0.69  | 10.8                                    | 0.1                 | 0.011      | 26.0                      | NSH                                    |
| Co.015   | 1   | (4) s  | 6.8         | 0.211                            | 0.063   | 1.2E-03             | 26.7                | 0.60  | 3.30                                    | 0.1                 | 0.011      |                           | slow CO startup (120 min)              |
|          | 2   | f      | 7.0         | 0.388                            | 0.135   | 2.3E-03             | 26.4                | 0.64  | 1.39                                    | 0.1                 | 0.011      |                           | NSH                                    |
| Co.016   | 1   | (4a) s | 5.2         | 0.161                            | 0.035   | 9.4E-04             | 33.6                | 0.56  | 6.85                                    |                     |            |                           | slow CO startup (30 min)               |
|          | 2   | f      | 5.1         | 0.291                            | 0.112   | 1.7E-03             | 25.1                | 0.68  | 2.48                                    | 0.1                 | 0.01       |                           | NSH                                    |

Table 5. Summary of Fixed Bed Reaction Results

(Continued)

| Catalyst | Run | Prep   | CO Conv. (%) | Rate g CH <sub>2</sub> /g cat/hr | R-C6+ g CH <sub>2</sub> /g cat/hr | R (Co) mol/mol Co/s | wt% CH <sub>4</sub> | alpha | C3-C5 Ole/Par | CO <sub>2</sub> (%) | g/g cat/hr | E act kcal/mol | Comment                                |
|----------|-----|--------|--------------|----------------------------------|-----------------------------------|---------------------|---------------------|-------|---------------|---------------------|------------|----------------|--|
| Co.017   | 2   | (1) s  | 4.6          | 0.139                            | 0.066                             | 8.2E-04             | 22.0                | 0.69  | 3.72          | 0.1                 | 0.012      |                | NS                                     |
| "        | 2a  | s      | 3.7          | 0.111                            | 0.043                             | 6.5E-04             | 25.9                | 0.64  | 4.19          | 0.1                 | 0.011      |                | CO:H <sub>2</sub> :Ar = 1:2:2          |
| "        | 2b  | s      | 3.4          | 0.102                            | 0.039                             | 6.0E-04             | 26.9                | 0.65  | 4.39          | 0.1                 | 0.013      |                | + H <sub>2</sub> O (eq. 7.5% CO conv.) |
| Co.018   | 2   | s      | 3.4          | 0.170                            | 0.068                             | 1.0E-03             | 27.2                | 0.69  | 2.24          | 0.1                 | 0.013      |                | NS                                     |
| "        | 2a  | s      | 2.7          | 0.133                            | 0.041                             | 7.8E-04             | 33.2                | 0.64  | 2.52          | 0.1                 | 0.011      |                | CO:H <sub>2</sub> :Ar = 1:2:2          |
| "        | 2b  | s      | 3.0          | 0.147                            | 0.045                             | 8.6E-04             | 33.6                | 0.63  | 2.41          | 0.1                 | 0.015      |                | + H <sub>2</sub> O (eq. 7.5% CO conv.) |
| "        | 3   | s      | 4.1          | 0.290                            | 0.086                             | 1.7E-03             | 30.0                | 0.62  | 2.34          | 0.1                 | 0.022      |                | NSH                                    |
| "        | 4   | s      | 3.6          | 0.340                            | 0.180                             | 2.0E-03             | 25.4                | 0.80  | 1.92          | 0.1                 | 0.041      |                | NSH, 10 atm                            |
| "        | 5   | f      | 7.5          | 0.470                            | 0.136                             | 2.8E-03             | 29.0                | 0.60  | 1.93          | 0.3                 | 0.055      |                | NSH                                    |
| "        | 6   | f      | 6.5          | 0.409                            | 0.147                             | 2.4E-03             | 24.6                | 0.64  | 2.42          |                     |            |                | NSH,S2"                                |
| Co.018Cl | 1   | (1)    | 4.5          | 0.320                            | 0.075                             | 1.9E-03             | 33.5                | 0.57  | 1.45          | 0.1                 | 0.018      |                | NSH                                    |
| Co.019   | 1   | (1)s   | 4.4          | 0.088                            | 0.047                             | 5.2E-04             | 15.2                | 0.74  | 7.61          |                     |            |                | NS                                     |
| "        | 2   | s      | 2.2          | 0.085                            | 0.046                             | 4.9E-03             | 18.9                | 0.73  | 8.59          | 0.3                 | 0.040      |                | NSH                                    |
| "        | 3   | s      | 3.4          | 0.142                            | 0.071                             | 8.3E-04             | 19.3                | 0.72  | 6.74          | 0.2                 | 0.025      |                | NSH                                    |
| "        | 3h  | f      | 16.7         | 0.172                            | 0.091                             | 1.0E-03             | 18.2                | 0.74  | 1.77          | 1.0                 | 0.034      |                | NSH, high conv. study                  |
| Co.020a  | 1   | (4) s  | 4.6          | 0.343                            | 0.085                             | 2.0E-03             | 33.5                | 0.57  | 1.70          | 0.1                 | 0.024      |                | NSH                                    |
| "        | 2   |        | 4.4          | 0.315                            | 0.086                             | 1.8E-03             | 31.2                | 0.59  | 1.96          | 0.1                 | 0.028      |                | NSH                                    |
| Co.020b  | 1   | (4b) s | 4.4          | 0.311                            | 0.084                             | 1.8E-03             | 31.7                | 0.58  | 1.81          | 0.2                 | 0.051      |                | NSH                                    |
| Co.020c  | 1   | (4a) s | 4.0          | 0.287                            | 0.083                             | 1.7E-03             | 30.4                | 0.60  | 2.38          | 0.1                 | 0.017      |                | NSH                                    |
| Co.021   | 1   | (6b)   | 3.4          | 0.109                            | 0.032                             | 6.4E-04             | 27.3                | 0.62  | 2.78          | 0.1                 | 0.012      | 28.2           | NSH                                    |
| "        | 2   |        | 3.6          | 0.114                            | 0.035                             | 6.7E-04             | 28.0                | 0.56  | 2.69          | 0.1                 | 0.012      |                | NSH                                    |
| Co.022   | 1   | (6c)   | 3.8          | 0.121                            | 0.037                             | 7.1E-04             | 26.7                | 0.55  | 2.91          | 0.1                 | 0.015      | 30.1           | NSH                                    |
| Co.023   | 1   | (6c)   | 3.8          | 0.123                            | 0.038                             | 7.2E-04             | 28.3                | 0.56  | 3.18          | 0.2                 | 0.023      | 29.5           | NSH                                    |
| Co.024   | 1   | (7)    | 5.1          | 0.165                            | 0.047                             | 9.7E-04             | 32.8                | 0.62  | 2.08          | 0.5                 | 0.055      |                | NSH                                    |
| "        | 2   |        | 5.7          | 0.182                            | 0.060                             | 1.1E-03             | 28.7                | 0.62  | 2.25          | 0.3                 | 0.035      |                | NSH                                    |
| Co.025   | 1   | (4)    | 5.0          | 0.160                            | 0.062                             | 9.4E-04             | 23.5                | 0.63  | 4.13          | 0.2                 | 0.019      | 26.9           | NSH                                    |
| Co.026   | 1   | (1)    | 4.4          | 0.129                            | 0.043                             | 5.0E-04             | 26.9                | 0.62  | 2.65          | 0.2                 | 0.022      |                | NSH                                    |
| "        | 1h  |        | 22.9         | 0.170                            | 0.072                             | 6.6E-04             | 22.6                | 0.67  | 0.64          | 1.5                 | 0.037      |                | NSH, high conv. study                  |
| Co.027   | 1   | (1)    | 4.2          | 0.169                            | 0.068                             | 6.6E-04             | 23.0                | 0.65  | 3.44          | 0.2                 | 0.026      |                | NSH                                    |
| "        | 1h  |        | 17.8         | 0.180                            | 0.085                             | 7.0E-04             | 19.4                | 0.71  | 0.96          | 1.2                 | 0.011      |                | NSH, high conv. study                  |
| Co.028   | 1   | (4)    | 3.8          | 0.146                            | 0.076                             | 5.7E-04             | 18.2                | 0.76  | 7.63          | 0.2                 | 0.031      |                | NSH                                    |
| "        | 2   |        | 4.3          | 0.167                            | 0.105                             | 6.5E-04             | 14.7                | 0.80  | 13.2          | 0.2                 | 0.030      | 28.4           | NSH                                    |
| "        | 3   |        | 4.5          | 0.206                            | 0.126                             | 8.0E-04             | 15.5                | 0.79  | 13.4          | 0.2                 | 0.037      |                | NSH, re-calc. in flow air              |
| Co.029   | 1   | (4)    | 2.8          | 0.173                            | 0.105                             | 6.7E-04             | 15.3                | 0.79  | 25.9          | 0.2                 | 0.026      |                | NSH                                    |
| Co.031   | 1   | (1)    | 1.9          | 0.060                            | 0.022                             | 3.5E-04             | 26.1                | 0.64  | 4.65          | 0.3                 | 0.031      |                | NSH                                    |
| Co.032   | 1   | (1)    | 3.3          | 0.183                            | 0.079                             | 1.1E-03             | 22.0                | 0.70  | 5.28          | 0.1                 | 0.022      |                | NSH                                    |
| Co.033   | 1   | (1)    | 1.3          | 0.073                            | 0.027                             | 4.3E-04             | 24.1                | 0.67  | 7.36          | 0.1                 | 0.020      |                | NSH                                    |
| Co.034   | 1   | (4)    | 5.0          | 0.275                            | 0.107                             | 1.6E-03             | 24.0                | 0.67  | 4.29          | 0.1                 | 0.018      |                | NSH                                    |
| Co.035   | 2   | (6)    | 4.6          | 0.147                            | 0.068                             | 8.6E-04             | 22.0                | 0.69  | 4.84          | 0.2                 | 0.019      |                | NSH                                    |
| Co.036   | 2   | (6c)   | 3.9          | 0.125                            | 0.045                             | 7.3E-04             | 28.9                | 0.67  | 4.20          | 0.1                 | 0.013      |                | NSH                                    |
| Co.037   | 1   | (4a)   | 2.7          | 0.053                            | 0.011                             | 5.2E-04             | 32.8                | 0.59  | 2.82          |                     |            |                | NSH                                    |
| Co.038   | 1   | (4a)   | 4.5          | 0.051                            | 0.018                             | 4.9E-04             | 25.1                | 0.68  | 4.63          | 0.3                 | 0.013      | 24.2           | NSH                                    |

Table 5. Summary of Fixed Bed Reaction Results

(Continued)

| Catalyst | Run | Prep  | CO Conv (%) | Rate g CH <sub>2</sub> /g cat/hr | R:Co+ g CH <sub>2</sub> /g cat/hr | R (Co) mol/mol Co/s | wt% CH <sub>4</sub> | alpha | C <sub>2</sub> -C <sub>5</sub> Ole/Par | CO <sub>2</sub> (%) g/g cat/hr | B:act kcal/mol | Comments |                              |
|----------|-----|-------|-------------|----------------------------------|-----------------------------------|---------------------|---------------------|-------|--|--------------------------------|----------------|----------|------------------------------|
| Co.039   | 1   | (5)   | 1.1         | 0.021                            | 0.004                             | 2.0E-04             | 57.2                | 0.64  | 0.84                                   | 0.2                            | 0.010          | 25.6     | NSH, cracking in the line    |
| "        | 2   |       | 3.9         | 0.049                            | 0.007                             | 4.7E-04             | 42.4                | 0.48  | 0.95                                   | 0.2                            | 0.009          |          | NSH                          |
| Co.040   | 1   | (4a)  | 1.9         | 0.037                            | 0.004                             | 3.7E-04             | 45.2                | 0.53  | 1.79                                   | 0.2                            | 0.007          |          | NSH                          |
| "        | 2   |       | 2.8         | 0.033                            | 0.004                             | 2.0E-04             | 44.6                | 0.46  | 1.12                                   | 0.2                            | 0.007          |          | NSH                          |
| Co.041   | 1   | (4)   | 3.5         | 0.136                            | 0.042                             | 8.0E-04             | 40.7                | 0.69  | 1.47                                   | 0.6                            | 0.079          |          | NSH, cracking in the line    |
| Co.043   | 1   | (4a)  | 4.6         | 0.104                            | 0.062                             | 6.1E-04             | 15.3                | 0.78  | 12.1                                   | 0.6                            | 0.048          |          | NSH, 2.5 hr on-stream        |
| "        | 2   |       | 3.7         | 0.078                            | 0.045                             | 4.6E-04             | 16.3                | 0.78  | 7.75                                   | 0.5                            | 0.035          | 35.1     | NSH                          |
| Co.047   | 1   | (4)   | 6.4         | 0.263                            | 0.128                             | 1.5E-03             | 21.3                | 0.73  | 7.20                                   | 0.3                            | 0.040          | 26.6     | NSH                          |
| Co.048   | 1   | (4)   | 3.5         | 0.162                            | 0.094                             | 9.5E-04             | 16.3                | 0.77  | 17.1                                   | 0.2                            | 0.027          | 28.0     | NSH                          |
| Co.049   | 1   | (4)   | 7.1         | 0.366                            | 0.153                             | 2.1E-03             | 23.4                | 0.69  | 5.63                                   | 0.3                            | 0.043          |          | NSH                          |
| "        | 2   |       | 6.0         | 0.388                            | 0.160                             | 2.3E-03             | 23.3                | 0.68  | 5.93                                   | 0.3                            | 0.056          |          | NSH, re-calcined in flow air |
| "        | 3   | f     | 6.7         | 0.410                            | 0.158                             | 2.4E-03             | 25.2                | 0.69  | 4.64                                   | 0.3                            | 0.059          |          | NSH                          |
| "        | 3h  | f     | 44.9        | 0.457                            | 0.178                             | 2.7E-03             | 26.5                | 0.67  | 0.67                                   | 3.8                            | 0.131          |          | NSH,HCS                      |
| Co.053   | 1   | (1)   | 7.0         | 0.408                            | 0.127                             | 2.4E-03             | 27.3                | 0.62  | 3.07                                   | 0.2                            | 0.049          |          | NSH                          |
| "        | 1h  |       | 28.5        | 0.417                            | 0.145                             | 2.4E-03             | 26.8                | 0.64  | 0.79                                   | 1.6                            | 0.081          |          | NSH, high conv. study        |
| "        | 2   | f     | 6.6         | 0.433                            | 0.174                             | 2.5E-03             | 23.4                | 0.63  | 2.35                                   | 0.2                            | 0.052          |          | NSH                          |
| "        | 3   |       | 7.8         | 0.453                            | 0.143                             | 2.7E-03             | 27.6                | 0.62  | 1.67                                   | 0.1                            | 0.027          |          | NSH                          |
| "        | 3h  |       | 31.5        | 0.457                            | 0.164                             | 2.7E-03             | 25.4                | 0.65  | 0.52                                   | 0.9                            | 0.044          |          | NSH, high conv. study        |
| "        | 4   | u     | 6.1         | 0.426                            | 0.142                             | 2.5E-03             | 28.3                | 0.65  | 1.41                                   | 0.2                            | 0.030          |          | NSH                          |
| Co.054   | 1   | (1)   | 1.2         | 0.041                            | 0.023                             | 2.4E-04             | 17.8                | 0.79  | 16.3                                   | 0.2                            | 0.014          |          | NSH                          |
| Co.055   | 1   | (1) f | 9.1         | 0.490                            | 0.159                             | 2.9E-03             | 28.9                | 0.63  | 1.31                                   | 0.2                            | 0.034          |          | NSH                          |
| "        | 2   | u     | 4.9         | 0.290                            | 0.105                             | 1.7E-03             | 25.7                | 0.65  | 1.49                                   | 0.2                            | 0.022          |          | NSH, T serge                 |
| "        | 3   | u     | 11.0        | 0.611                            | 0.193                             | 3.6E-03             | 29.4                | 0.60  | 1.02                                   | 0.2                            | 0.033          |          | NSH                          |
| CAL.01   | 1   | (1)   | 4.8         | 0.181                            | 0.068                             | 1.1E-03             | 24.4                | 0.66  | 5.86                                   | 0.3                            | 0.036          |          | NSH, temp. surge (startup)   |
| "        | 2   |       | 7.4         | 0.491                            | 0.142                             | 2.9E-03             | 28.7                | 0.61  | 2.06                                   | 0.4                            | 0.083          |          | NSH                          |
| "        | 3   |       | 2.5         | 0.164                            | 0.053                             | 9.6E-04             | 25.6                | 0.63  | 4.97                                   | 0.2                            | 0.052          |          | NSH, HT, re-reduced          |
| "        | 4   |       | 7.3         | 0.483                            | 0.103                             | 2.8E-03             | 35.8                | 0.57  | 1.21                                   | 0.2                            | 0.098          |          | NSH, HT, re-calcined         |
| CAL.02   | 1   | (1)   | 6.1         | 0.379                            | 0.121                             | 2.2E-03             | 27.3                | 0.63  | 3.04                                   | 0.2                            | 0.040          |          | NSH                          |
| CAL.03   | 1   | (1)   | 7.3         | 0.459                            | 0.133                             | 2.7E-03             | 29.0                | 0.60  | 2.59                                   | 0.2                            | 0.048          |          | NSH                          |
| CAL.04   | 1   | (1)   | 4.1         | 0.234                            | 0.123                             | 1.4E-03             | 19.1                | 0.71  | 8.98                                   | 0.2                            | 0.026          |          | NSH                          |
| CAL.05   | 1   | (1)   | 4.7         | 0.274                            | 0.134                             | 1.6E-03             | 20.8                | 0.75  | 6.85                                   | 0.2                            | 0.026          |          | NSH                          |
| CAL.06   | 1   | (1)   | 5.8         | 0.267                            | 0.135                             | 1.6E-03             | 20.7                | 0.74  | 5.50                                   | 0.2                            | 0.021          |          | NSH                          |
| CoW.01   | 1   | (4a)  | 1.2         | 0.016                            | 0.003                             | 9.5E-05             | 30.2                | 0.58  | 3.78                                   | 0.2                            | 0.006          |          | NSH, reduced at 230 °C       |
| "        | 1   |       | 0.5         | 0.007                            | 0.001                             | 3.4E-05             | 42.1                | 0.51  | 0.95                                   | 0.1                            | 0.003          |          | NSH, re-reduced at 350 °C    |
| "        | 2   |       | 0.2         | 0.006                            | 0.000                             | 3.4E-05             | 40.9                | 0.51  | 11.3                                   | 0.1                            | 0.006          |          | NSH, reduced at 350 °C       |
| CoW.02   | 1   | (4a)  | 0.3         | 0.009                            | 0.002                             | 5.4E-05             | 25.6                | 0.60  | 4.94                                   | 0.1                            | 0.008          |          | NSH, reduced at 230 °C       |
| CoW.03   | 1   | (4a)  |             |                                  |                                   |                     |                     |       |  |                                |                |          | NSH, negligible activity     |
| CoW.04   | 1   | (4a)  |             |                                  |                                   |                     |                     |       |  |                                |                |          | S2, negligible activity      |
| CoW.05   | 1   | (4c)  | 2.8         | 0.035                            | 0.006                             | 2.1E-04             | 33.1                | 0.55  | 0.66                                   | 0.2                            | 0.008          |          | S2, reduced at 350 °C        |
| "        | 2   | u     | 4.7         | 0.058                            | 0.011                             | 3.4E-04             | 34.8                | 0.50  | 0.85                                   | 0.2                            | 0.009          |          | S2                           |
| CoW.06   | 1   | (4a)  | 3.3         | 0.041                            | 0.012                             | 2.4E-04             | 26.0                | 0.59  | 2.21                                   | 0.2                            | 0.007          |          | S2, reduced at 250 °C        |
| "        | 2   | u     | 2.6         | 0.032                            | 0.004                             | 1.9E-04             | 39.3                | 0.46  | 0.99                                   | 0.3                            | 0.012          |          | S2                           |
| "        | 2   |       | 17.0        | 0.207                            | 0.004                             | 1.2E-03             | 68.7                | 0.32  | 0.37                                   | 1.7                            | 0.07           |          | S2, rxn. at 280 °C           |
| UOP      | 1   | (1)   | 7.8         | 0.298                            | 0.104                             | 1.7E-03             | 22.6                | 0.61  | 1.29                                   | 0.2                            | 0.023          |          | NSH                          |

## Table 5. Summary of Fixed Bed Reaction Results

(Continued)

All the reactions are carried out at 220 deg. C , 1 atm , and with H<sub>2</sub>:CO=2

All the catalysts are reduced and passivated before loading into reactor, then rereduced in-situ

All catalysts are calcined at UPARC (static) : except where specified in prep. column

s : calcined at Pitt, static

f : calcined at Pitt, flow

u : uncalcined

In some cases, "re-calcined" means calcination of the calcined catalyst from UPARC for a longer period

(1) single step, aqueous incipient wetness

(2) Two step impregnation: aqueous incipient wetness of Co + acetone/ethanol impregnation of Ru, La

(3) three step, with calcination after each step, acetone incipient wetness

(4) two step, aqueous incipient wetness, (a) intermediate calcination, (b) intermediate reduction, (c) intermediate calcination 750 °C

(5) single step, acetone impregnation

(6) single step, aqueous kneading, (a) = 150%, (b) = 110% of pore volume

(6c) aqueous kneading for Co, calcined, then aqueous incipient wetness for Zr

(7) Two step impregnation :organic incipient wetness + aqueous incipient wetness

\* New startup

\*\* New startup, heated inlet

" was done in the second system

~ Davison Grade 59 Silica, if not specified, S refers Davison Grade 952 Silica

# corrected overheated line on 3/6/1994

Cl chlorinated (see Co.018Cl)

Table 6

DATE: 09/30/94

COMPARISON OF CONVERSION AND SELECTIVITY OF  
METHANE REDUCTION CATALYSTS AT STARTUP CONDITIONS

| Period No. | Temp.   | Pres.     | H <sub>2</sub> /CO Ratio |            |                  |                 |                  |                  |          |           |                |                        |       |
|------------|---------|-----------|--------------------------|------------|------------------|-----------------|------------------|------------------|----------|-----------|----------------|------------------------|-------|
| -----      | -----   | -----     | -----                    |            |                  |                 |                  |                  |          |           |                |                        |       |
| 1          | 240 C   | 450psi    | 2.0                      |            |                  |                 |                  |                  |          |           |                |                        |       |
| Run No     | Cat. No | Cat. wt,g | Conv. %                  | Prod. Rate | Selectivities    |                 |                  |                  | Alpha GC | Alpha Liq | Catalyst Prom% | Composit. Metal% Suppt |       |
| -----      | -----   | -----     | -----                    | -----      | %CH <sub>4</sub> | %C <sub>2</sub> | %C <sub>3+</sub> | %CO <sub>2</sub> | -----    | -----     | -----          | -----                  | ----- |
| M-4        | SBCR    |           |                          |            |                  |                 |                  |                  |          |           |                |                        |       |
| 3          | Co.002  | 15.2      | 29.4                     | 1.42       | 17.3             | 2.3             | 79.4             | 1.01             | 0.73     | 0.82      | 1.0La          | 0.43Ru                 | Al    |
| 14         | BlendA  | 20.0      | 29.3                     | 1.41       | 10.1             | 1.6             | 86.3             | 2.02             | 0.77     | 0.86      | 5.0Cu          | 4.0Cr                  | Al    |
| 15         | Co.034  | 13.0      | 27.5                     | 1.54       | 10.4             | 1.6             | 87.2             | 0.80             | 0.78     | 0.84      | 8.5Zr          | 0.0                    | Al    |
| 16         | CAL.04  | 16.1      | 26.3                     | 1.34       | 7.1              | 1.3             | 90.3             | 1.38             | 0.80     | 0.85      | 0.3K           | 0.5Ru                  | Al    |
| 17         | CAL.08  | 15.0      | 16.9                     | 0.93       | 5.9              | 0.8             | 91.7             | 1.36             | 0.82     | 0.88      | 0.3K           | 0.5Ru                  | Al    |
| 18         | CAL.07  | 15.0      | 20.6                     | 1.03       | 7.7              | 1.2             | 90.0             | 1.09             | 0.81     | 0.88      | 0.3K           | 0.5Ru                  | Al    |
| 19         | CAL.05  | 15.7      | 26.6                     | 1.22       | 7.3              | 1.3             | 90.2             | 1.19             | 0.82     | 0.86      | 0.3K           | 0.5Ru                  | Al    |
| 20         | CAL.06  | 15.0      | 5.6                      | 0.26       | 3.1              | 2.1             | 92.0             | 2.86             | 0.80     | 0.90      | 0.3K           | 0.5Ru                  | Al    |
| 21         | CO.004  | 15.9      | 39.3                     | 1.80       | 15.2             | 2.3             | 81.2             | 1.38             | 0.74     | 0.80      | 1.0La          | 0.43Ru                 | Al    |

Note: Catalysts for all runs except Run 8 screened thru 150 x 400 mesh.  
Blend A contains 15.0 gm of Cat. No. Co.005 plus 5.0 gm of Cat. No. WGS.03.  
Cat. Nos. CAL.07 and CAL.08 were prereduced and wax coated.  
H<sub>2</sub> to CO ratio for Runs 16 and 17 was 1.76/1.0.  
Cat. No. CAL.06 was H<sub>2</sub> reduced and air stabilized.



Table 7

DATE: 09/30/94

COMPARISON OF CONVERSION AND SELECTIVITY OF  
CALISICAT PRODUCED CATALYSTS AT STARTUP CONDITIONS

| Period No. | Temp. | Pres.  | H2/CO Ratio |
|------------|-------|--------|-------------|
| 1          | 240 C | 450psi | 2.0         |

- a) Total flow is ca. 15 L/min. STP, or 3 cm/sec linear gas flow.  
Flows: N2-563 SLH, H2-225 SLH, CO-112.5 SLH  
b) Conversion is total CO conversion over the period (%).  
c) Conversion and selectivities are calculated using N2 as an internal standard in the GC analysis of the offgas.  
d) Prod. rate: Rate for production of total hydrocarbons C1+ (kg/kg cat., hr).  
e) Alpha: Based on GC analysis of offgas and liquid product.

| Run No   | Cat. No | Cat. wt, g | Conv. % | Prod. Rate | Selectivities |     |      |      | Alpha GC | Alpha Liq | Catalyst Prom% | Composit. Metal% | Suppt |
|----------|---------|------------|---------|------------|---------------|-----|------|------|----------|-----------|----------------|------------------|-------|
|          |         |            |         |            | %CH4          | %C2 | %C3+ | %CO2 |          |           |                |                  |       |
| M-4 SBCR |         |            |         |            |               |     |      |      |          |           |                |                  |       |
| 10       | CAL.02  | 15.9       | 34.5    | 1.58       | 12.9          | 2.0 | 83.9 | 1.23 | 0.75     | 0.81      | 0.0            | 0.5Ru            | Al    |
| 11       | CAL.03  | 15.6       | 29.7    | 1.39       | 13.5          | 2.1 | 83.4 | 1.08 | 0.74     | 0.86      | 0.0            | 0.5Ru            | Al    |
| 16       | CAL.04  | 16.1       | 26.3    | 1.34       | 7.1           | 1.3 | 90.3 | 1.38 | 0.80     | 0.85      | 0.3K           | 0.5Ru            | Al    |
| 17       | CAL.08  | 15.0       | 16.9    | 0.93       | 5.9           | 0.8 | 91.7 | 1.36 | 0.82     | 0.88      | 0.3K           | 0.5Ru            | Al    |
| 18       | CAL.07  | 15.0       | 20.6    | 1.03       | 7.7           | 1.2 | 90.0 | 1.09 | 0.81     | 0.88      | 0.3K           | 0.5Ru            | Al    |
| 19       | CAL.05  | 15.7       | 26.6    | 1.22       | 7.3           | 1.3 | 90.2 | 1.19 | 0.82     | 0.86      | 0.3K           | 0.5Ru            | Al    |
| 20       | CAL.06  | 15.0       | 5.6     | 0.26       | 3.1           | 2.1 | 92.0 | 2.86 | 0.80     | 0.90      | 0.3K           | 0.5Ru            | Al    |
| M-3 SBCR |         |            |         |            |               |     |      |      |          |           |                |                  |       |
| 12       | CO.018  | 15.6       | 33.8    | 1.56       | 9.7           | 2.0 | 86.6 | 1.68 | 0.75     | 0.85      | 0.0            | 0.5Ru            | Al    |
| 23       | CO.047  | 15.8       | 28.2    | 1.29       | 7.7           | 1.5 | 89.3 | 1.47 | 0.79     | 0.84      | 0.3K           | 0.5Ru            | Al    |

Note: Catalysts for all runs except Runs 17, 18, & 20 screened thru 150 x 400 mesh.  
Catalyst CAL.04 was calcined in air; Catalyst CAL.05 processed in N2.  
Cat. No. CAL.06 was H2 reduced and air stabilized before charging.  
Cat. Nos. CAL.08 and CAL.07 were prereduced and wax coated. CAL.08 was calcined in N2; CAL.07 was reduced in H2 without prior calcination.  
H2 to CO ratio for Runs 16 and 17 was 1.76/1.  
Cat. Nos. Co.018 & Co.047 were prepared by Pitt. Shown for comparison.

Table 8

HIGH SYNGAS CONVERSION TEST: RUN 29-M3

| <u>Period No.</u> | <u>Temp.</u><br>°C | <u>Pressure</u><br>psi | <u>H<sub>2</sub>/CO</u><br><u>Ratio</u> | <u>CO Conv.</u><br>% | <u>Prod. Rate</u><br>KgC <sub>1</sub> +/<br>Kg, Hr | <u>Selectivities</u>    |                        |
|-------------------|--------------------|------------------------|---|----------------------|--|-------------------------|------------------------|
|                   |                    |                        |   |                      |  | <u>% CH<sub>4</sub></u> | <u>%C<sub>3</sub>+</u> |
| 1                 | 240                | 450                    | 2.0                                     | 42.0                 | 1.19   | 15.8                    | 79.5                   |
| 2                 | 220                | 450                    | 2.0                                     | 20.5                 | 0.59   | 5.7                     | 89.0                   |
| 3                 | 240                | 450                    | 2.0                                     | 39.2                 | 1.13   | 15.8                    | 79.2                   |
| 4                 | 240                | 450                    | 1.0                                     | 18.4                 | 0.79   | 5.3                     | 90.6                   |
| 5                 | 240                | 600                    | 1.0                                     | 19.7                 | 0.85   | 4.6                     | 92.9                   |
| 6                 | 240                | 600                    | 2.0                                     | 40.8                 | 1.17   | 14.8                    | 81.4                   |
| 7                 | 240                | 450                    | 2.0 (1)                                 | 31.5                 | 0.45   | 21.0                    | 72.2                   |
| 8                 | 260                | 450                    | 2.0 (1)                                 | 31.0                 | 0.44   | 36.4                    | 52.6                   |
| 9                 | 240                | 450                    | 2.0                                     | 18.0                 | 0.55   | 23.0                    | 72.1                   |
| 10                | 240                | 450                    | 2.0 (2)                                 | 21.6                 | 0.98   | 14.4                    | 82.4                   |

## Notes:

(1) Feed gas composition: N<sub>2</sub>-80%, H<sub>2</sub>-13.5%, CO-6.5%(2) Feed gas composition: N<sub>2</sub>-40%, H<sub>2</sub>-40%, CO-20%All other periods: N<sub>2</sub>-60%, H<sub>2</sub>-33%, CO-17%

Table 9

## SBCR CATALYST EXTRACTIONS

| Catalyst No.<br>(Support) | Run No. | Charge                             |                                    | Recovered<br>(Oxidized)<br>wt, gm | Charge                                |                                       | Recovered                         |                                       | Reduction<br>in Particle<br>Size,<br>% |
|---------------------------|---------|------------------------------------|------------------------------------|-----------------------------------|---------------------------------------|---------------------------------------|-----------------------------------|---------------------------------------|--|
|                           |         | (H <sub>2</sub> Reduced)<br>wt, gm | (H <sub>2</sub> Reduced)<br>wt, gm |                                   | Particle<br>Size<br>at 10%<br>microns | Particle<br>Size<br>at 10%<br>microns | Particle<br>Size, mvd,<br>microns | Particle<br>Size<br>at 10%<br>microns |  |
| Co.001 (Al)               | 1       | 14.5                               | 14.5                               | 12.63                             | 42.02                                 | 24.25                                 | 41.06                             | 23.62                                 | 2.3                                    |
| Co.001 (Al)               | 2       | 16.5                               | 16.5                               | 15.18                             | 42.02                                 | 24.25                                 | 40.09                             | 22.86                                 | 4.6                                    |
| Co.005 (Al)               | 3       | 19.5                               | 19.5                               | 18.41                             | 85.66                                 | 30.46                                 | 64.53                             | 31.27                                 | 24.7                                   |
| Co.002 (Al)               | 4       | 20.1                               | 20.1                               | 20.4                              | 73.61                                 | 32.86                                 | 71.19                             | 33.50                                 | 3.3                                    |
| Co.003 (Al)               | 5       | 15.1                               | 15.1                               | 14.54                             | 76.81                                 | 34.40                                 | 72.57                             | 34.25                                 | 5.5                                    |
| Co.011 (Si)               | 6       | 15.3                               | 15.3                               | 16.03                             | 107.09 (1)                            | 53.19                                 | 94.02                             | 40.44                                 | 12.2                                   |
| Co.012 (Si)               | 7       | 15.8                               | 15.8                               | 14.73                             | 105.22 (1)                            | 51.33                                 | 101.37                            | 44.44                                 | 3.7                                    |
| Co.015 (Al)               | 8       | 15.0                               | 15.0                               | 15.52                             | 75.63 (1)                             | 33.15                                 | 74.68                             | 32.68                                 | 1.3                                    |
| Co.011 (Si)               | 9       | 15.0                               | 15.0                               | 15.92                             | 79.88 (2)                             | 48.31                                 | 72.44                             | 36.31                                 | 9.3                                    |
| Co.016 (Al)               | 10      | 15.5                               | 15.5                               | 15.72                             | 64.60 (2)                             | 32.16                                 | 64.38                             | 32.78                                 | 0.3                                    |
| Co.017 (Al)               | 11      | 15.0                               | 15.0                               | 15.09                             | 73.14 (3)                             | 36.29                                 | 69.95                             | 35.62                                 | 4.4                                    |
| Co.018 (Al)               | 12      | 15.6                               | 15.6                               | 16.97                             | 62.89 (3)                             | 32.56                                 | 68.16                             | 34.74                                 | ---                                    |
| Co.016 (Al)               | 13      | 14.7                               | 14.7                               | 13.30                             | 67.69 (3)                             | 35.05                                 | 64.79                             | 34.20                                 | 4.3                                    |
| Co.019 (Si)               | 14      | 15.2                               | 15.2                               | 15.00                             | 92.63 (3)                             | 42.73                                 | 84.39                             | 36.52                                 | 8.9                                    |
| Co.005 (Al)               | 15      | 14.7                               | 14.7                               | 15.30                             | 80.62 (3)                             | 45.07                                 | 71.90                             | 36.79                                 | 10.8                                   |
| Co.002 (Al)               | 16      | 15.4                               | 15.4                               | 14.98                             | 73.61 (3)                             | 32.86                                 | 67.62                             | 32.67                                 | 8.1                                    |
| Co.002 (Al)               | 1 (M4)  | 15.0                               | 15.0                               | 11.00                             | 73.61 (3)                             | 32.86                                 | 66.26                             | 35.75                                 | 10.0                                   |
| Co.025 (Si)               | 17      | 15.6                               | 15.6                               | 14.60                             | 87.19 (3)                             | 47.35                                 | 78.31                             | 36.27                                 | 10.2                                   |
| Co.004 (Al)               | 18      | 15.0                               | 15.0                               | 15.25                             | 78.12 (3)                             | 44.15                                 | 66.86                             | 33.83                                 | 14.4                                   |
| Co.021 (Si)               | 19      | 15.6                               | 15.6                               | 16.06                             | 86.18 (3)                             | 46.54                                 | 77.95                             | 35.59                                 | 9.55                                   |
| Co.041 (Si)               | 20      | 15.8                               | 15.8                               | 17.02                             | 87.48 (3)                             | 49.26                                 | 68.74                             | 26.72                                 | 17.0                                   |
| Co.021 (Si)               | 3 (M4)  | 15.2                               | 15.2                               | 15.1                              | 73.61 (3)                             | 24.46                                 | 71.38                             | 32.33                                 | 3.03                                   |
| Co.024 (Al)               | 4 (M4)  | 15.4                               | 15.4                               | (4)                               | ---                                   | ---                                   | ---                               | ---                                   | ---                                    |
| Co.035 (Si)               | 5 (M4)  | 15.4                               | 15.4                               | 14.08                             | 89.14 (3)                             | 49.29                                 | 74.76                             | 31.67                                 | 19.2                                   |
| Co.028 (Al)               | 6 (M4)  | 15.4                               | 15.4                               | 15.7                              | 80.83 (3)                             | 44.81                                 | 71.83                             | 36.34                                 | 11.1                                   |

(1) Screened through 100x400 mesh screens. (2) Screened through 170x400 mesh screens.  
 (3) Screened through 150x400 mesh screens. (4) Did not filter, too waxy

Note: The charge weights are in the hydrogen-reduced state and the recovered weights are in the oxidized state. The particle size is reported as the mean volume diameter (mvd) as measured by a Microtrac particle size analyzer.

Table 9  
(Continued)

SBCR CATALYST EXTRACTIONS

| Catalyst No.<br>(Support) | Run No. | Charge                             |                                    | Recovered<br>(Oxidized)<br>wt, gm | Charge                                |                                       | Recovered                         |                                       | Reduction<br>in Particle<br>Size,<br>% |
|---------------------------|---------|------------------------------------|------------------------------------|-----------------------------------|---------------------------------------|---------------------------------------|-----------------------------------|---------------------------------------|--|
|                           |         | (H <sub>2</sub> Reduced)<br>wt, gm | (H <sub>2</sub> Reduced)<br>wt, gm |                                   | Particle<br>Size<br>at 10%<br>microns | Particle<br>Size<br>at 10%<br>microns | Particle<br>Size, mvd.<br>microns | Particle<br>Size<br>at 10%<br>microns |  |
| Co.014 (Ti)               | 21      | 15.1                               | 111.70 (1)                         | 15.1                              | 32.65                                 | 106.66                                | 33.49                             | 4.51                                  |  |
| Co.040 (Ti)               | 22      | 14.5                               | (5)                                | 8.5                               | (5)                                   | 130.40                                | 51.40                             | --                                    |  |
| Co.047 (Al)               | 23      | 15.8                               | 83.42 (3)                          | 16.95                             | 44.41                                 | 76.46                                 | 39.02                             | 8.34                                  |  |
| Co.049 (Al)               | 24      | 15.6                               | 81.3 (3)                           | 16.54                             | 42.39                                 | 70.11                                 | 35.87                             | 13.8                                  |  |
| Co.047 (Al)               | 25      | 15.6                               | 79.36 (3)                          | 16.69                             | 41.72                                 | 69.09                                 | 36.16                             | 12.94                                 |  |
| Co.031 (Al)               | 26      | 15.9                               | 75.48 (3)                          | 15.37                             | 38.34                                 | 68.42                                 | 35.39                             | 9.35                                  |  |
| Co.029 (Al)               | 27      | 15.9                               | 76.39 (3)                          | 18.28                             | 39.56                                 | 68.72                                 | 36.04                             | 10.04                                 |  |
| Co.043 (Si)               | 7 (M4)  | 15.4                               | (5)                                | 15.1                              | (5)                                   | 85.51                                 | 40.20                             | --                                    |  |
| Co.006 (Ti)               | 8 (M4)  | 15.0                               | 121.38 (1)                         | 9.09                              | 52.80                                 | 99.31                                 | 38.62                             | 18.2                                  |  |
| Co.048 (Si)               | 9 (M4)  | 15.4                               | 82.40 (3)                          | 15.85                             | 40.94                                 | 70.69                                 | 25.53                             | 14.2                                  |  |
| CAL.02 (Al)               | 10 (M4) | 15.9                               | 74.40 (3)                          | 16.42                             | 37.79                                 | 65.89                                 | 34.14                             | 11.44                                 |  |
| Co.053 (Al)               | 12 (M4) | 15.9                               | 82.02 (3)                          | 16.95                             | 43.10                                 | 69.53                                 | 35.76                             | 15.23                                 |  |
| Co.054 (Si)               | 13 (M4) | 15.7                               | 101.17 (3)                         | 17.09                             | 57.54                                 | 82.37                                 | 37.89                             | 18.58                                 |  |
| Blend A (Al)              | 14 (M4) | 20.0                               | 86.23 (3)                          | 14.61                             | 46.36                                 | 77.43                                 | 42.81                             | 10.21                                 |  |
| Co.034 (Al)               | 15 (M4) | 13.0                               | (5)                                | 9.52                              | (5)                                   | 88.26                                 | 45.28                             | --                                    |  |
| CAL.04 (Al)               | 16 (M4) | 16.1                               | 71.54 (3)                          | 21.78                             | 36.28                                 | 66.50                                 | 32.82                             | 7.05                                  |  |
| CAL.08 (Al)               | 17 (M4) | 15.0 (6)                           | 63.12                              | 14.27                             | 25.60                                 | 62.80                                 | 26.03                             | 0.51                                  |  |
| CAL.07 (Al)               | 18 (M4) | 14.5 (6)                           | --                                 | 13.37                             | --                                    | 67.02                                 | 28.12                             | --                                    |  |
| CAL.05 (Al)               | 19 (M4) | 15.7                               | 72.75                              | 5.62                              | 36.90                                 | 71.62                                 | 36.55                             | 1.55                                  |  |
| CAL.06 (Al)               | 20 (M4) | 15.0                               | 72.62                              | (7)                               | 37.33                                 | --                                    | --                                | --                                    |  |
| Co.053 (Al)               | 29 (M3) | 25.2                               | 75.63                              | 25.8                              | 40.80                                 | 71.09                                 | 38.20                             | 6.00                                  |  |
| Co.004 (Al)               | 21 (M4) | 15.9                               | 81.38                              | 15.04                             | 41.82                                 | 72.88                                 | 37.61                             | 10.44                                 |  |

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- (1) Screened through 100x400 mesh screens.
  - (3) Screened through 150x400 mesh screens.
  - (5) Insufficient feed sample.
  - (6) Wax coated.
  - (7) Lost catalyst.