

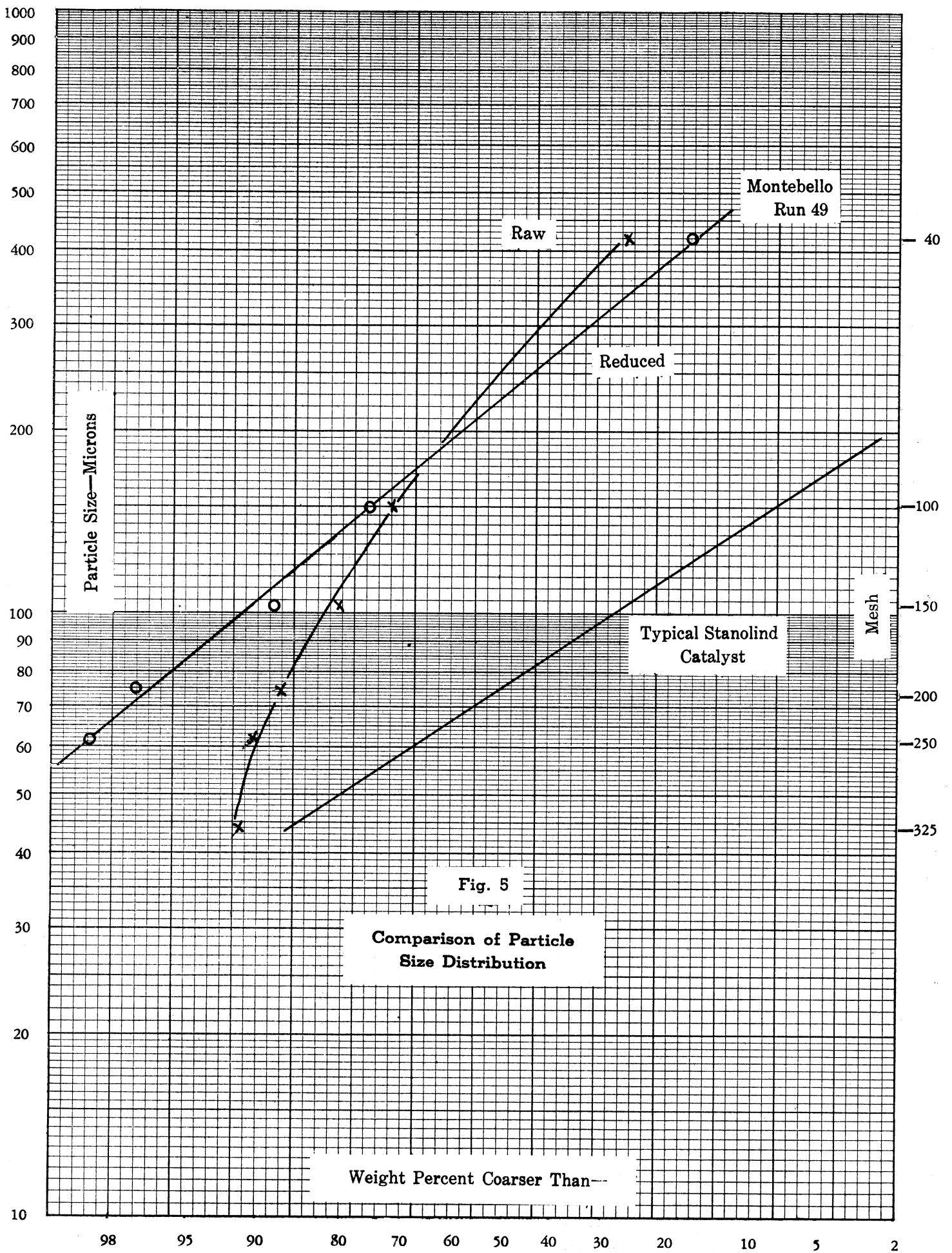
III. SUMMARY OF OPERATIONS

A. Catalyst Reduction

The original charge of catalyst was reduced in place by circulating cylinder hydrogen at 200 psig and 700°F. at a linear velocity of one foot per second through the reactor system until water production had virtually ceased. The weight of water recovered after 62 hours corresponded to 95 per cent reduction. This was later confirmed by X-ray diffraction analysis.

The progress of reduction is shown in the opposite Figure 4 which indicates that the rate of water production was low at the beginning (presumably because of the low temperature) and was also low at the end as the reaction approached completion. During the central portion the rate of water production was 18.2 lbs. per hour. This indicates that the rate is controlled by hydrogen availability.

During the reduction the original charge of 2558 lbs. of oxide yielded 595 lbs. of water and 265 lbs. of fines were carried out of the reactor leaving 1750 lbs. of reduced catalyst.



The screen analyses of raw and reduced catalyst showed:

| <u>Screen Analyses of Raw and Reduced Catalysts</u> | | | | <u>Raw</u> | <u>Reduced</u> |
|---------------------------------------------------------|---------------|-----|---------|------------|----------------|
| Wt. Per Cent on | | | | | |
| | 40 mesh, | 419 | microns | 26.3 | 16.3 |
| | 100 " | 150 | " | 46.0 | 59.9 |
| | 150 " | 105 | " | 8.3 | 12.4 |
| | 200 " | 74 | " | 7.3 | 8.6 |
| | 250 " | 62 | " | 2.6 | 1.0 |
| | 325 " | 44 | " | 0.8 | 1.0 |
| | Through 325 " | | | 8.7 | 0.8 |

If it is assumed that all of the fines were through 325 mesh, the reduced catalyst screen analysis can be corrected for the loss and then shows:

| | | |
|-----------------|---------------|------|
| Wt. Per Cent on | | |
| | 40 mesh | 14.7 |
| | 100 " | 53.9 |
| | 150 " | 11.2 |
| | 200 " | 7.7 |
| | 250 " | 0.9 |
| | 325 " | 0.9 |
| | Through 325 " | 10.7 |

Comparing this corrected analysis with the raw feed the indicated changes in the various fractions are:

| | <u>Raw</u> | <u>Corrected Product</u> | <u>Change</u> |
|----------------|------------|------------------------------|---------------|
| 0-40 mesh | 26.3 | 14.7 | -11.6 |
| 40-100 mesh | 46.0 | 53.9 | + 7.9 |
| 100-150 mesh | 8.3 | 11.2 | + 2.9 |
| 150-200 mesh | 7.3 | 7.7 | + 0.4 |
| 200-250 mesh | 2.6 | 0.9 | - 1.7 |
| 250-325 mesh | 0.8 | 0.9 | + 0.1 |
| below 325 mesh | 8.7 | 10.7 | + 2.0 |

This shows that the principal changes on reduction were the fracture of about half the 0-40 mesh particles into the 40-150 mesh range and the elutriation of most of the particles below 325 mesh in size. This effect is illustrated by the opposite Figure 5 where the logarithm of particle size has been plotted against a

normal probability function. The raw catalyst shows a curved distribution on this plot, whereas the reduced catalyst shows a linear relationship. This linear form was retained throughout the run and is characteristic of all operations on the Montebello unit. It is believed to be inherent to an elutriating system.

This inability of a bed of coarsely ground catalyst to retain very fine particles is believed to explain the absence of "bug-dusting" on the Montebello unit. During Run 49, for example, the catalyst removed at the end of the run did not differ greatly from the freshly reduced catalyst as shown by the following table:

| <u>Screen Analysis</u> | <u>Weight Per Cent</u> | |
|------------------------------------|------------------------|------------------|
| | <u>Initial</u> | <u>500 Hours</u> |
| On 40 mesh | 16.3 | 14.1 |
| 100 " | 59.9 | 66.6 |
| 150 " | 12.4 | 10.9 |
| 200 " | 8.6 | 4.8 |
| 250 " | 1.0 | 1.4 |
| 325 " | 1.0 | 1.0 |
| Thru 325 " | 0.8 | 1.2 |
| <u>Bulk Density - Lbs./Cu. Ft.</u> | | |
| In Reactor | 154 | 121 |
| Lab. Aerated Density | 159 | 122 |

Over this period there was no material decline in heat transfer coefficient, indicating that the excellent scouring action of the freshly reduced catalyst was retained throughout the run.

A typical particle size distribution for the Stanolind operations at Tulsa is also shown in Figure 5. This unit has commonly experienced difficulty with "defluidization", presumably as a result of the finer catalyst grind which is used. This finer catalyst has a much less effective scrubbing action and is much more susceptible to wetting than the coarser catalyst used at Montebello and it is therefore not surprising that the unit should show greater difficulty in operation.

B. Start-Up Procedure

The synthesis unit was put on-stream in the same way as in Runs 46 and 48. After reduction was finished, water was fed to the boiler system and the circulation of hot hydrogen continued until a water level was established and pressure built up to 500 psig. This gave a catalyst temperature of 470°F. Fresh feed was then introduced and preheat reduced to hold the bed in the range of 650°F. The above procedure requires less than two hours.

C. Synthesis Operation

The system was lined out at 420 psig inlet pressure, 650°F. bed temperature, 15 thousand standard cubic feet per hour of fresh feed, and a recycle ratio of 1/1. This gave an inlet velocity of 1 foot per second. These conditions were maintained for 340 hours with a catalyst addition rate of 50 lbs. per day.

The recycle rate was then increased to give a recycle ratio of 1.5/1 and the run continued for an additional 156 hours. Other conditions remained unchanged.

At the end of this time, the fresh feed rate was reduced to 10 thousand standard cubic feet per hour and the recycle ratio returned to 1/1 for an additional 30 hours. The data obtained in this last short period are not reliable due to low weight recovery (71 per cent) caused by a leak in the product condenser.

This leak apparently started at about 460 hours when weight recovery fell from 95 to 85 per cent. Inspection of the

TABLE I
SUMMARY OF DATA - RUN 49

400 psig, 650°F., Alan Wood - 1.2% K₂O basis Fe

| Test Period | Hours on Stream | Average Cat. Age Hours | Rates MCFH | | Inlet Vel. ft/sec. | Bed Depth Ft. | Space Vel. v/hr/v | Conversion % of H ₂ +CO Fed | Selectivity C ₃ + : C ₁ + | Yield of C ₃ + #/MCF(1) | Chemicals from water #/MCF |
|-------------|-----------------|------------------------|------------|---------|--------------------|---------------|-------------------|----------------------------------------|-------------------------------------------------|------------------------------------|----------------------------|
| | | | Feed | Recycle | | | | | | | |
| A | 16 | 16 | 14.8 | 14.2 | 0.96 | 20 | 1000 | 86.2 | 81.8 | 9.46 | 0.73 |
| B | 34 | 34 | 15.0 | 15.5 | 1.01 | 20.8 | 1055 | 83.7 | 84.3 | 9.57 | 0.83 |
| C | 59 | 59 | 15.3 | 15.6 | 1.02 | 19.5 | 1140 | 80.4 | 82.6 | 8.36 | 0.72 |
| D | 79 | 79 | 15.5 | 15.4 | 1.02 | 20.3 | 1120 | 81.1 | 83.1 | 8.88 | 0.64 |
| E | 104 | 104 | 15.2 | 15.3 | 1.04 | 20.4 | 1088 | 82.1 | 81.5 | 8.34 | 0.94 |
| F | 128 | 120 | 15.5 | 15.4 | 1.08 | 22.7 | 997 | 78.5 | 83.4 | 8.52 | 0.91 |
| G | 152 | 135 | 15.6 | 15.3 | 1.09 | 22.8 | 994 | 79.0 | 83.7 | 8.52 | 0.89 |
| H | 176 | 151 | 15.7 | 15.4 | 1.10 | 21.1 | 1094 | 79.2 | 82.8 | 8.22 | 0.86 |
| I | 200 | 174 | 15.8 | 15.7 | 1.07 | 20.5 | 1122 | 77.4 | 81.1 | 8.33 | 0.83 |
| K | 221 | 186 | 15.1 | 14.8 | 0.97 | 21.3 | 1037 | 77.8 | 80.2 | 8.06 | 0.83 |
| L | 245 | 202 | 15.3 | 15.7 | 1.01 | 21.6 | 1033 | 77.9 | 81.8 | 8.63 | 0.89 |
| M | 269 | 217 | 15.1 | 15.5 | 0.99 | 20.3 | 1085 | 77.7 | 82.1 | 8.50 | 0.86 |
| N | 293 | 231 | 15.5 | 15.7 | 1.01 | 20.1 | 1127 | 78.6 | 82.7 | 8.47 | 0.89 |
| O | 317 | 244 | 15.4 | 15.7 | 1.04 | 20.7 | 1083 | 77.5 | 82.0 | 8.17 | 0.87 |
| P | 341 | 257 | 15.2 | 15.8 | 1.01 | 19.1 | 1163 | 76.5 | 81.9 | 8.13 | 0.89 |
| 49-1 Avg. | | 192 | 15.42 | 15.5 | 1.037 | 21.02 | 1074 | 78.01 | 82.26 | 8.369 | 0.871 |
| Q | 365 | 274 | 15.6 | 23.2 | 1.24 | 19.8 | 1142 | 77.1 | 82.1 | 8.44 | 0.90 |
| R | 389 | 285 | 15.8 | 24.5 | 1.39 | 18.5 | 1244 | 76.3 | 83.6 | 8.27 | 0.96 |
| S | 413 | 295 | 15.8 | 23.3 | 1.36 | 18.4 | 1256 | 76.7 | 83.1 | 8.32 | 0.88 |
| T | 437 | 304 | 15.5 | 23.1 | 1.35 | 18.7 | 1203 | 76.5 | 82.4 | 8.43 | 0.91 |
| U | 461 | 312 | 16.2 | 23.6 | 1.41 | 18.8 | 1252 | 76.5 | 82.4 | 7.96 | 0.95 |
| V | 473 | 305 | 16.1 | 23.5 | 1.41 | 19.8 | 1185 | 75.6 | 83.3 | 8.09 | 0.92 |
| W | 497 | 312 | 16.1 | 23.4 | 1.39 | 19.2 | 1219 | 76.1 | 82.6 | 8.02 | 0.94 |
| 49-2 Avg. | | 298 | 15.87 | 23.51 | 1.364 | 19.03 | 1214 | 76.40 | 82.79 | 8.208 | 0.921 |
| X | 521 | 316 | 10.6 | 11.2 | 0.79 | 17.2 | 894 | 83.0 | 85.4 | 9.80 | 1.26 |
| Y | 528 | 301 | 10.7 | 10.6 | 0.77 | 18.2 | 856 | 83.2 | 85.1 | 9.57 | 1.32 |

(1) Includes Chemicals from Water

tube bundle showed that the leak was caused by the penetration of a pit from the water side of one of the tubes at the hot end of the condenser. This tube bundle had been in service from the time the Montebello unit was first started - about 3 years total service. The bundle was carbon steel and there was no measurable corrosion from the vapor side. The pitting on the cooling water side was confined to a few tubes at the hot end with a single case where the pit penetrated through the tube wall.