

III. RESULTS AND DISCUSSIONOperations - Run 58

The reduction of new CM&S catalyst for Run 58 was conducted in the reactor with a hydrogen flow of 1 ft./sec. velocity through the bed which was maintained at 670°-760°F for 44 hours. The initial charge of catalyst was 2500 pounds, but the carryover of fines was 424 pounds during the first 18 hours of reduction. This made it necessary to load an additional 400 pounds of unreduced catalyst to the reactor. The 88 pounds of water made during the first 18 hours of reduction was considerably less than normal (usually about 170 pounds of water from 2500 pounds of mill scale or magnetite after 18 hours at temperatures above 650°F). The reduction was so slow that the supply of hydrogen was depleted before the reduction was finished, because of normal loss of hydrogen from blowing down cyclones, water accumulators, etc. It was necessary to cut in the synthesis gas prematurely rather than shut down and run the risk of plugging the reactor inlet. The total catalyst charged was 2900 pounds, the catalyst carried over was 871 pounds, and the water removed was 290.5 pounds. The raw material was analyzed as 67.1 per cent iron and the partially reduced catalyst was 78.3 per cent iron.

Run 58 lasted only 83 hours because a leak developed in the product condenser tube bundle. It was planned to reduce the catalyst more completely and continue the run after the condenser was repaired, but the bottom of the reactor became plugged while standing idle. Therefore the catalyst was removed from the unit and discarded.

Operations - Run 59

The catalyst for Run 59 had been previously used in ammonia synthesis at Trail, B. C. before shipment to Montebello Laboratory. Analysis of the material as received showed 84 per cent iron but there was no indication of its being pyrophoric. The initial charge of catalyst to the reactor was 2514 pounds, but more than 700 pounds blew over during the ensuing reduction with hydrogen. Since the catalyst was already essentially reduced, further reduction in the reactor at 750° to 775°F produced only 37 pounds of water.

During the initial 178 hours of the run, the trend was toward a decline in catalyst density and inventory. The catalyst bed temperatures increased daily even though no preheat was used and the steam pressure in the cooling tubes was kept lower than normal.

Run 59 was interrupted after 178 hours (Period H) by a failure of the recycle compressor engine. This caused the reactor inlet to plug, and it was necessary to remove the catalyst (988 pounds) to clear the plug. The catalyst was recharged and reduced with hydrogen until water production ceased. This second treatment produced 138 pounds of water.

After the catalyst was reduced again, the catalyst density and inventory were increased by periodic additions of reduced catalyst. The bed temperatures were gradually brought down to the normal 650°F level.

There was another shutdown after 781 hours, between periods HH and II, in order to conduct a scheduled insurance

inspection of the plant boiler. During this time hydrogen was circulated through the bed at 660°F and 1 ft./sec. inlet velocity. The run was terminated voluntarily after 843 hours.

Operations - Runs 60 and 61

These two runs have been grouped together because the operating conditions were very similar. The catalysts were obtained in separate shipments, but after grinding and reduction, the particle size distributions were identical. The average particle size of the reduced catalyst was 185 microns for Runs 60 and 61 in contrast to 350 microns or more in the other runs.

In both Runs 60 and 61 the initial charge was 2500 pounds, the carryover of fines was 800 pounds, and the reduced catalyst analyzed 87 per cent iron. The reduction time was 104 hours in Run 60 and 90 hours in Run 61.

After only 25 hours on stream a slug of water carried over into the reactor system from the synthesis gas scrubber because of a failure in the water level control system. The bed temperatures dropped from 650°F to 450°F, but the catalyst meters gave no indication of loss of "fluidization", that is, the meter fluctuations continued at the same frequency and amplitude.

The run was discontinued long enough to reduce the catalyst again but after the second reduction the yields were never as high again as in Period A.

Run 61 was a duplicate of Run 60, without the water carryover, but the yields were higher. There was no catalyst addition during the run. The density of the fluidized catalyst was 104 lbs./cu.ft. at the end of Period A and declined after that. This was comparable to the 106 lbs./cu.ft. density at end of Period A

in Run 60, but after the contamination with water and subsequent reduction in Run 60, the density increased to the 160 lbs./cu.ft. level.

As in Run 59 the low catalyst density in Run 61 was accompanied by difficulty in controlling the bed temperatures except to a much greater extent. After 239 hours Run 61 was terminated when the upper bed temperatures were in the 900°-950°F range and out of control. When the catalyst was removed from the reactor, it was found to contain many loosely consolidated lumps which were easily crumbled with the fingers. There was little change in particle size during the run, but the density declined because the carbon content increased from about 5 per cent at the beginning of the run to about 20 per cent at the end.

Operations - Run 62

With the thought that the instability of Run 61 may have been the result of high catalyst activity and low inlet velocity, Run 62 was made with 15 MSCFH fresh feed rate instead of the 10 MSCFH rate used in Runs 60 and 61.

After Run 61 the reactor steam system had been cleaned with inhibited hydrochloric acid to eliminate the possibility of boiler scale in the cooling tubes causing difficulty in controlling the catalyst bed temperature.

Run 62 was made with fresh CM&S catalyst ground to pass through a 14-mesh sieve. The initial charge to the reactor was 2500 pounds. The reduction required 90 hours, the catalyst carry-over was 624 pounds, and 572 pounds of water was formed.

After 542 hours of stable operations, the run was voluntarily terminated.

Table I
OPERATING CONDITIONS AND YIELD DATA SUMMARY

Period	Hours on Stream	Average Catalyst Age, Hrs	Rates, MSCFH		Bed Depth, Feet	Catalyst Size 50% Point, Microns	Space Velocity v/hr/v	Reactor Back Pressure PSIG(1)	Conversion H ₂ + CO Per Cent	Selectivity, C ₃ +C ₁ + Per Cent	Activity Index(2)	C ₃ +, lbs/MCF H ₂ +CO Fed	Barrels/Day Basis Brownsville Design Feed Rate
			Fresh Feed	Recycle									
58-0	0-93	47	15.31	16.67	15.2	350	1563	361	71.16	79.33	21.35	7.60	5595
59-0	0-86	43	16.96	14.62	17.0	547	1472	375	87.13	82.50	34.16	9.24	7316
59-1	86-178	132	17.30	15.00	15.4	494	1657	372	84.84	81.68	33.35	8.66	6811
59-2	178-291	166	15.60	15.47	12.0	323	1813	375	84.65	79.78	34.66	8.96	7068
59-3	291-445	211	13.30	16.18	15.7	331	1225	375	84.51	80.06	28.35	8.60	6674
59-4	445-541	228	14.58	15.83	20.2	320	1057	372	85.64	81.18	27.40	9.14	7196
59-5	541-661	303	14.44	14.47	19.7	385	1072	365	85.03	80.38	27.00	9.12	7084
59-6	661-781	376	14.64	14.41	19.6	345	1095	361	86.45	78.60	28.73	9.19	7154
59-7	781-843	432	14.58	14.74	19.1	430	1114	373	86.53	79.12	29.06	9.38	7288
60-0	0-88	40	11.17	16.55	11.9	180 ⁽³⁾	1431	369	75.46	84.07	23.08	8.89	6924
60-1	88-160	97	11.21	16.95	13.5	--	1222	366	73.56	84.91	20.20	8.56	6557
61-0	0-89	45	11.45	14.50	22.6	180	754	382	92.08	85.78	27.84	10.75	8553
61-1	89-239	164	11.27	15.49	24.2	168	692	378	85.29	83.71	21.90	9.49	7426
62-0	0-94	44	16.69	16.57	17.8	340	1385	375	77.32	83.27	23.98	8.66	6739
62-1	94-206	138	15.63	16.62	19.2	--	1192	373	75.48	83.15	21.08	8.58	6632
62-2	206-303	243	15.70	17.00	18.9	--	1226	372	73.39	83.06	20.13	8.08	6395
62-3	303-399	339	15.44	17.33	17.6	--	1309	371	68.77	81.66	18.29	7.43	5815
62-4	399-543	416	15.56	17.03	15.1	--	1513	370	71.32	82.45	21.10	7.74	6141

(1) Reactor inlet pressure was usually 25 to 40 psi greater than the back pressure.

(2) Activity Index = $\sqrt{v}/hr/v \log\left(\frac{100}{100 - \text{conversion}}\right)$

(3) Estimated.