TABLE 2.—*Bench-scale, trickle-flow experiments with cobalt*

Experiment No.	FT-85c	FT-87b	FT-88a	FT-88b	FT-88c	FT-88d
Duration, hr.	91.8	81.1	88.3	77.2	101.9	114.5
Catalyst:						
Total age, hr.	483	486	382	382	382	382
Volume, cu. ft.	←— 0.053 —→					
Mode of operation	←— Countercurrent —→			←— Cocurrent —→		Countercurrent
Oil:						
Boiling range at 1 atm., ° C.	130–150	110–140	110–125	110–125	110–125	110–125
Flow, ml. per hr.	250	260	400	2,110	1,910	800
Synthesis gas:						
Ratio in fresh gas, H_2:CO	1.67	1.94	2.13	2.08	2.08	2.13
S. V. H.[1] (settled bed)	99	64	121	122	100	104
Flow, cu. ft. per hr. (S. T. P.)	6.50	3.33	6.61	6.57	5.38	5.60
Contraction, vol.-percent[2]	60.1	48.5	48.4	50.2	45.9	54.9
Conversion, vol.-percent H_2+CO	61.5	49.8	48.6	50.7	46.4	54.8
Usage ratio, H_2:CO	1.87	2.17	2.21	2.07	2.00	2.14
Pressure, average, p. s. i. g.	57.3	66.7	68.9	71.4	67.5	84.0
Ave. temperature, ° C.	198	184	173	175	173	189
Yields, specific, gm. per m^3 C_1+C_2	40.7	83.9	51.9	36.0	46.3	57.4

[1] See Glossary (p. 70).
[2] Measured after passage through liquid-nitrogen traps so that gas was virtually free of hydrocarbons.

Comparison shows that neither countercurrent nor cocurrent trickle-flow operation afforded conversion as high, or gaseous hydrocarbon production as low, as fixed-dry-bed operations.

The comparatively close approach of the usage ratio to a value of 2.0, ideal for the synthesis reaction catalyzed by cobalt, is a good indication of favorable product distribution, that is, preponderance of liquids and solids. High yields of methane would have resulted in a usage ratio closer to 3.0. No direct measurements of liquids and solids production were undertaken for this series, because early in the work it became evident that the procedures for separating coolant from product would be too laborious and complex to be justified at this scale of operation.

The data in table 2 indicate a slight but positive advantage, with respect to controlling the production of low-molecular-weight hydrocarbons, when gas flow was cocurrent with the liquid. When operated in this manner the liquid coolant input was about 10 times the quantity required in countercurrent operation, since there was virtually no liquid reflux.

In general, process performance obtained in this series of experiments met expectations rather well. Under countercurrent flow conditions, operation was continuous and trouble free. Radial temperature gradients were negligible, and longitudinal gradients did not exceed 5° C. Cocurrent operation was usually difficult to control; slight variations in either coolant or gas flow initiated wide fluctuations in temperature, and consequently fluctuations in gas conversion.

Catalyst life was apparently not adversely affected, either chemically or physically, by the comparatively severe treatment inherent to the process. When discharged, the pellets were found to be intact.

PILOT PLANTS[3]

THREE-INCH-DIAMETER REACTOR

OBJECTIVE

To investigate the principle of direct internal cooling on a larger scale and make a fairly complete process study, a pilot plant with a 3-inch-diameter reactor and an allowable bed depth of 8 feet was built. It was completed in March 1945. It was designed for operation with countercurrent or cocurrent flow. The 8-foot bed would permit a more complete, accurate study of the longitudinal temperature gradients than the 1-foot bed that had been used previously.

The program followed in the pilot plants can be divided into three parts that are separately reviewed in the remaining portion of this report.

1. Trickle-flow experiments. This group comprises experiments 1 to 5-E, employing pelleted cobalt catalyst, and experiments 7 to 9-B, employing precipitated iron.
2. Submerged fixed-bed experiments.

(a) Preliminary tests, comprising 2 tests made with pelleted cobalt (experiments 5-F and 6) and 1 with

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[3] The pilot plants were operated by A. J. Forney, R. M. Jimeson, T. H. Ross, W. P. Haynes, J. E. Tosh, G. E. Johnson, M. Fenton, H. Chiswick, F. J. Schoenewerk, L. Chollinet, and D. Bernstein. Construction of the units was supervised by W. H. Williams.

Chemical analyses were performed by the staff of the Analytical Chemistry Section of the Synthetic Fuels Research Branch under the supervision of R. D. Hinkel, M. R. Rundle, and W. K. Pfister. X-ray and magnetic analyses of the catalysts were conducted by L. J. E. Hofer, W. C. Peebles, and E. M. Cohn; and mass spectrometric and infrared analyses were made by R. A. Friedel, A. G. Sharkey, and J. L. Shultz.

precipitated iron (experiment 9–C). All of these tests were brief.

(b) Experiments 10 to 18, employing the synthetic ammonia-type iron catalyst.

3. Expanded-bed experiments comprising runs 19 to 26, also employing the synthetic ammonia-type iron catalyst.

TRICKLE-FLOW EXPERIMENTS

APPARATUS

The converter consisted of a 110-inch length of seamless, 3-inch, Schedule 40 pipe with 300-pound ring-joint flanges at each end (fig. 3). Twenty-two thermocouples in ¼- by 6-inch pipe along the length of the reactor were spaced 4 inches apart. Bare thermocouple junctions extending to different points along the radius of the bed entered ports made pressure-tight by gasketed flanges. A 0.5-inch plate, perforated with ¼-inch holes, at the bottom of the reactor supported the catalyst charge and Aloxite packing. Gas and liquid entries were provided in the top and bottom heads as shown. Because the unit was planned for service up to 300 p. s. i. g., welded connections were employed where practical. To compensate for heat losses by radiation, the converter was wound with Nichrome wire that allowed a maximum heat input of 2 kilowatts.

The schematic flowsheet of the pilot plant for the initial operation in March 1946 is shown in figure 4. Several changes in the system were made as required. Experiments 2-E to 5–E and 7 to 9–B were conducted in the cocurrent downward manner shown in figure 4, while experiments 1 and 2–A to 2-D

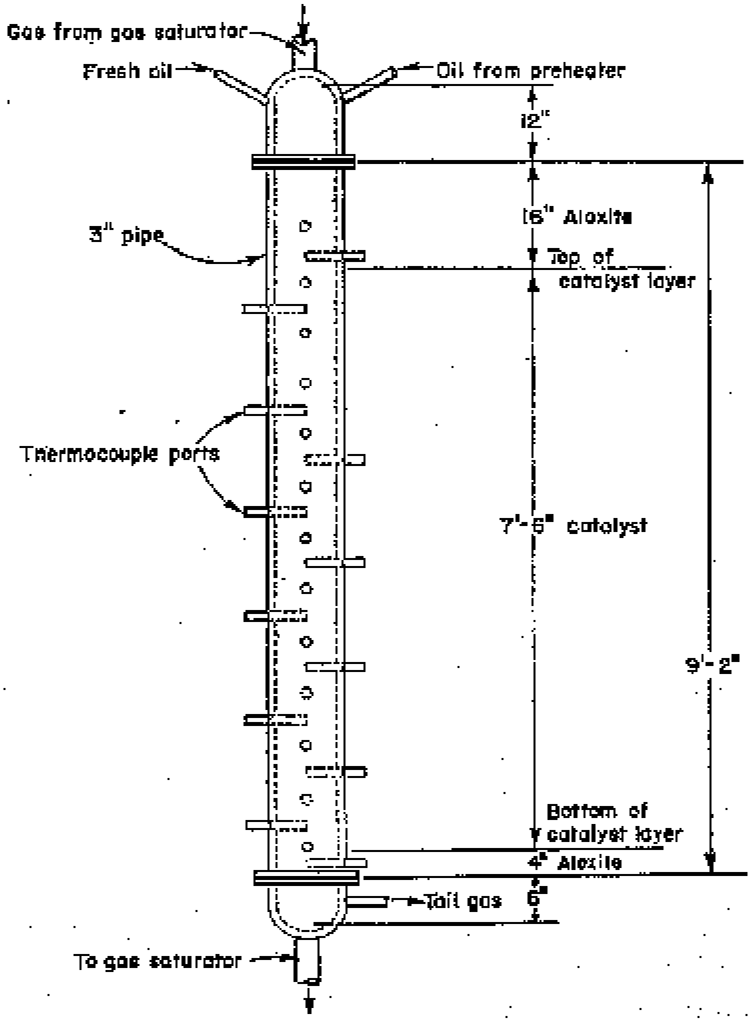

FIGURE 3.—Eight-Foot Converter for Trickle-Flow Process.

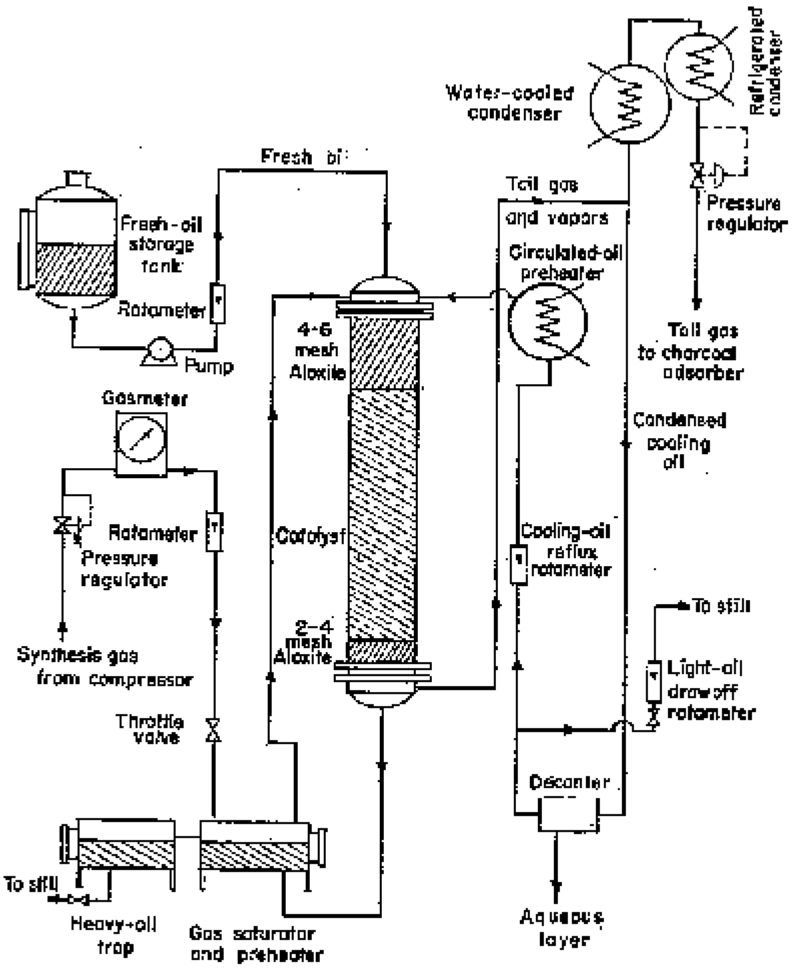

Figure 4.—Trickle-Flow Process.

were operated with the gas entering at the bottom of the reactor and leaving at the top.

To operate in cocurrent downward flow both gas and coolant liquid were introduced at the top of the converter. As shown on the flowsheet, the gas stream (at operating pressure) passed first through the gas saturator, where it was both preheated and equilibrated with the vapor of the coolant, then into the converter. Gases leaving the converter were conducted to atmosphere via a water-cooled condenser, operated at about 40° C., a refrigerated condenser operated at about 5° C., a back-pressure regulator, and an activated-charcoal tower.

Heavier, higher boiling products of the synthesis, along with any excess of cooling liquid, flowed by gravity back to the gas saturator, which was maintained at constant level by periodic withdrawals of the oil. Water, condensed cooling fluid, and part of the products of synthesis collected in the decanter. The aqueous fraction was continuously removed through a float-controlled valve. The oil that collected in this part of the system was removed as required, and the remainder returned by gravity to the converter through the circulating oil preheater. Toward the end of run 5 a pump was installed in this oil line to insure a positive flow at all times. Further recovery of the cooling fluid and lighter, lower boiling products was effected in the refrigerated condenser. These products were withdrawn regularly. All C_3 and C_4 hydrocarbons and such of the higher molecular weight products as remained in the gas stream after it had passed through the refrigerated condenser were recovered in the charcoal tower. For countercurrent operation (experiments 1, 2-A, 2-B, 2-C, 2-D) the cooling oil flow was the same as just described, but gas was sent from the saturator to the bottom of the reactor.

RAW MATERIALS

SYNTHESIS GAS

Synthesis gas was produced as reported under Bench-Scale, Trickle-Flow Investigations (p. 9).

CATALYST

Catalysts employed in this phase of the development were either promoted and kieselguhr-supported cobalt, formed into ⅛- by ⅛-inch pellets, or unpromoted and unsupported iron, prepared by precipitation as the hydrated oxide, and used as granules or as ⅛- by ⅛-inch pellets.

COOLING OIL

Cuts of a commercially obtainable naphtha, APCO thinner (Anderson-Pritchard Co.), were used as the fresh coolant. A fraction boiling at 122° to 133° C. was used for the cobalt, but a cut with a higher boiling range (190° to 205° C.) was required for the iron catalysts because the operating temperatures were higher. These oils were not desulfurized, as the oils previously used in the bench-scale trickle-flow investigation had been. They contained 0.018 weight-percent sulfur, but this amount of sulfur did not appear to have any detrimental effect upon the catalyst.

OPERATION

The two types of catalysts behaved quite differently during synthesis and required different preliminary treatments to secure maximum productivity. Precipitated iron was readily activated for the synthesis by controlled treatment with synthesis gas. Cobalt required carefully conducted prereduction with hydrogen, followed by an equally rigid schedule of induction with synthesis gas. A detailed description of the operations with cobalt will be given first, then the modifications required for iron will be explained.

To minimize breakage of the relatively soft cobalt-kieselguhr pellets and prevent excessively dense packing, a special method of charging the catalyst was employed. After a 4-inch layer of 2- to 4-mesh Aloxite was deposited on the perforated support plate that was covered with a 12-mesh screen, catalyst equivalent to a 4-inch bed height was gently lowered into place. A chromel-alumel thermocouple was inserted through the well in the reactor wall. This process was repeated until the bed height was 90 inches. Finally, a 1-inch layer of 4- to 6-mesh Aloxite was placed on top of the catalyst. A metal cartridge 24 inches long and containing 16 inches of 4- to 6-mesh Aloxite to help distribute the liquid was suspended from the top of the converter.

The reduction procedure was conducted at atmospheric pressure. To begin the reduction the temperature was raised to 360° C. as rapidly as possible, while a hydrogen flow of about 10 cu. ft. per hour was maintained. At 360° C. the flow of hydrogen was increased to 200 to 400 cu. ft. per hour, equivalent to an hourly space velocity of 500 to 1,000, and these conditions were maintained for 4 hours. The flow was then reduced to about 5 cu. ft. per hr. and the temperature to 150° C.

Before synthesis the storage tank for fresh oil, the gas saturator, and the preheater for the recycled oil were filled to proper levels with fresh cooling oil. Hydrogen was replaced by nitrogen flowing at about 20 cu. ft. per hr., with the back-pressure regulator adjusted to maintain a pressure of about 40 p. s. i. g. To saturate the catalyst, fresh oil was pumped to the converter at a rate of about 1 to 2 gal. per hr. for at least 1 hour. At the same time heat was applied to the gas saturator and converter to keep the system at 150° C. In concurrent operations the flow of nitrogen and the elevated temperature were sufficient to cause part of the oil to vaporize and pass to the condenser with the nitrogen. The condensate flowed from there to the decanter and returned through the reflux rotameter to the top of the converter. As the system was now equilibrated, nitrogen was replaced by $2H_2+1CO$ gas flowing at an hourly space velocity of 100. Because of the sensitivity of the freshly reduced catalyst, operating conditions during the initial or induction period of the synthesis were carefully controlled. The temperature was slowly raised from 150° C. by increasing the pressure in small increments by adjusting the back-pressure regulator. Usually the temperature was maintained below 175° C., with the gas contraction held to a maximum of 50 percent for the first 48 hours. A 5° C. rise to 180° C. was permitted during the next 24 hours, after which induction was considered complete, and the temperature was adjusted as required to secure maximum productivity. By this time necessary adjustments had been made in the heat input to the saturator and reactor walls and in the rate at which the fresh oil was pumped to insure steady-state thermal conditions in the system. Light- and heavy-oil products were discharged periodically and distilled in batches. The portion boiling in the range of the cooling oil was added to the oil-storage tank.

To terminate an experiment the gas flow was stopped, and virtually all heat input to the system was cut off. The pressure was reduced to atmospheric, hydrogen was passed through at a low rate to flush the lines, and all liquids were drained after the reactor temperature had dropped below 150° C. When the temperature had dropped below 100° C. after several hours of additional flushing with hydrogen, the system was flooded with an inert gas (carbon dioxide or nitrogen); and, in instances where another run was planned with the same catalyst charge, the system was maintained at a slight positive gage pressure to prevent leakage of air to the catalyst. If the catalyst were to be discarded or stored it was discharged into gastight containers equipped for maintaining a positive pressure of an inert gas. Discharge to the plant atmosphere would have been hazardous because of the pyrophoric nature of the material.

Experiments 7, 8, 9–A, and 9–B were conducted with precipitated iron; cocurrent, trickle-type operation was used. Pelleted or granular iron was charged in the manner described for cobalt. No prereduction was required, but induction was carried out with $1H_2+1CO$ at an hourly space velocity of 100 for 24 hours. During induction the temperature generally varied from 230° to 250° C., and contraction was rather arbitrarily limited to a maximum of 40 percent. The operating pressure required to obtain a given temperature depended upon the boiling range of the cooling oil.

After induction, temperature and pressure were adjusted to achieve a desired conversion. Procedures for operation, shutdown, and catalyst discharge were similar to those described for synthesis with cobalt.

RESULTS

Operating and yield data for selected periods during experiments 1 to 5–E with cobalt catalysts are given in table 3. Periods were selected to represent certain types of operation. For example, 2–A and 2–D were countercurrent runs, the former an induction and the latter a production run at an hourly space velocity of about 100.

TABLE 3.—*Pilot-plant experiments with cobalt*

Experiment No.	2–A	2–D	2–F	3–B	3–C	3–E	3–G	5–B	5–C	5–D
Duration..............hr..	58.5	92.3	217.6	106.1	73.6	62.3	216.5	87.5	98	117
Mode of operation..............	Countercurrent, trickle flow		Cocurrent, trickle flow							
Catalyst dimensions, diameter×length..in..	⅛×⅛ cyl			3/16×⅛ cyl.				3/16×0.230 cyl		
Volume (settled)..............cu. ft..	0.405	0.405	0.405	0.385	0.385	0.385	0.385	0.385	0.250	0.280
Oil:										
Boiling range at 1 atm..............° C..	122–133	122–133	122–133	122–133	122–133	122–133	128–133	125–134	126–133	140–150
Fresh makeup flow..............gal. per hr..	0.28	0.13	0.10	0.14	0.19	0.23	0.16	0.05	0.05	0.08
Recycle rate..............do..	5.35	5.4	5.0	2.4	5.4	7.7	6.1	7.7	9.6	6.0
Synthesis gas:										
Ratio in fresh gas, H_2:CO..............	2.16	2.17	1.83	1.89	1.94	1.90	1.97	1.96	2.04	2.04
S. V. H. (settled bed)..............	93	93	103	93	102	250	281	102	250	438
Conversion..............vol.-percent H_2+CO..	41.6	77.6	71.8	67.8	65.8	48.6	47.7	68.7	45.4	35.6
Usage ratio, H_2:CO..............	1.92	1.81	1.93	2.20	2.18	2.10	2.58	2.18	2.09	2.45
Pressure:										
Average..............p. s. i. g..	83	93	100	85	150	210	160	63	119	163
Differential..............in. H_2O..	4.3	4.8	0.6	0.4	0.7	2.3	1.6	2.2	4.5	3.6
Temperature, ° C.:										
Average..............	187	189	183	184	184	190	195	193	192	201
Maximum..............	178	197	201	205	207	215	217	202	210	221
Yields, specific, gm. per m.³										
CH_4..............	65.1	49.8	22.2	44.5	33.1	36.7	28.3	36.4	39.8	60.5
C_2H_6..............	3.4	7.7	5.0	2.3	3.4	6.1	9.4	8.5	5.3	7.4
C_3H_8..............	17.6	14.6	11.8	10.9	10.6	17.3	9.6	22.9	4.8	17.2
C_4H_{10}..............	5.8	10.1	7.6	11.2	10.6	10.5	12.9	14.5	6.2	11.5
C_5+—calculated..............	184.5	150.8	173.2	156.4	168.5	185.2	170.8	179.2	161.1	160.1

None of the trickle-flow experiments after 2–D was made with countercurrent gas-oil flow. Except for direction of gas-oil flow, experiment 2–F was made under conditions comparable with 2–D. Parts B, C, E, and G of experiment 3 illustrate operation at space velocities varying from 93 to 281; runs 5–B, C, and D at hourly space velocities of 102 and 438 under otherwise similar conditions.

Typical inductions for pelleted and granular precipitated iron are illustrated by the data from experiments 7–A and 8–A in table 4. The information from experiments 7–B and C is typical for steady-state operation in a trickle-flow system at an hourly space velocity of 100. Results from experiments 7–D and 7–E, 8–B, and 9–A and 9–B are not shown, as steady conditions were not maintained during these tests either because studies of operating variables were being conducted or because of operating difficulties.

DISCUSSION

Contrary to the performance of the bench-scale unit, the early experiments of the pilot plant demonstrated quite positively that, in trickle-flow operation, the synthesis was controlled more easily when gas and coolant flowed cocurrently downward rather than when they flowed countercurrently. Thus, in experiment 2–F (table 3) the activity was essentially the same as in 2–D, but the specific yield of C_1 plus C_2 decreased significantly. This result

TABLE 4.—*Pilot-plant, trickle-flow experiments with precipitated iron*

[Mode of operation, cocurrent]

Experiment No.	7-A	7-B	7-C	8-A
Duration ... hr.	49	86.8	94.5	70
Catalyst:				
Dimensions, diameter × length ... in.	3¼×34 cyl.			3-6-mesh gran.
Volume (settled) ... cu. ft.	0.385	0.385	0.385	0.336
Oil:				
Boiling range at 1 atm. ° C.	196–205	170–185	170–185	195–205
Fresh makeup flow gal. per hr.	0.39	0.05	0.07	0.10
Recycle rate ... do.	2.8	4.6	4.1	4.2
Synthesis gas:				
Ratio in fresh gas, H_2:CO	0.98	1.08	0.97	0.95
S. V. H. (settled bed)	96.8	93.7	95.0	100.3
CO_2-free contraction vol.-percent	49.0	55.6	54.0	53.6
Conversion vol.-percent H_2+CO	80.8	80.0	89.6	81.4
Usage ratio, H_2:CO	0.84	0.90	0.57	0.56
Pressure:				
Average ... p. s. i. g.	82	129	130	87
Differential ... in. H_2O	5.0	2.5	2.2	3.2
Temperature, ° C.:				
Average	265	267	257	254
Maximum	280	270	279	264
Yields, specific, gm. per m.³:				
CH_4	18.7	16.7	19.2	([1])
C_2H_4—C_2H_6	15.9	14.7	18.8	([1])
C_3H_6	([1])	12.2	4.9	([1])
C_3H_8	([1])	11.7	14.8	([1])
C_4H_8	([1])	3.8	0.5	([1])
C_4H_{10}	([1])	7.0	8.4	([1])
C_5+—calculated	([1])	177.6	181.9	([1])
Material balance percent recovery	([1])	99.4	97.7	([1])

[1] Data not available.

confirmed trends observed in the bench-scale work. Comparison of the pressure drops through the catalyst bed in these experiments shows the substantial decrease brought about in 2-F by this change in flow. Probably the most significant advantage gained was elimination of the flooding that had occurred at relatively low flow rates (an hourly space velocity slightly exceeding 200) in countercurrent operation, and that had caused hot-spots at the bottom of the bed near the gas entry.

A plot of temperature, conversion, and methane yield along the converter during cocurrent downflow operation in experiment 3-D (fig. 5) shows that the upper 2 feet of the reactor served as a preheating zone. Below this level the reaction rate was fairly steady. Good temperature control was maintained along the catalyst bed except for a hot spot at the gas and liquid entry near the top of the bed; this difficulty was especially troublesome at space velocities above 300. Returning the oil from the overhead condenser to the top of the reactor by means of a pump in experiment 5-D, rather than merely by gravity flow, improved the distribution of liquid over the cross section and gave better control of the temperature at this area. Consequently, the flow of fresh gas was increased to an hourly space velocity of about 440 in experiment 5-D and to 600 in experiment 5-E (table 5). Temperature fluctuations near the top of the bed were again encountered, especially when the hourly space velocity exceeded 450. It appeared that this gas flow and a space-time yield of C_3+ hydrocarbons of 600 kg. per m.³ of catalyst per day were the maximum for this type of operation.

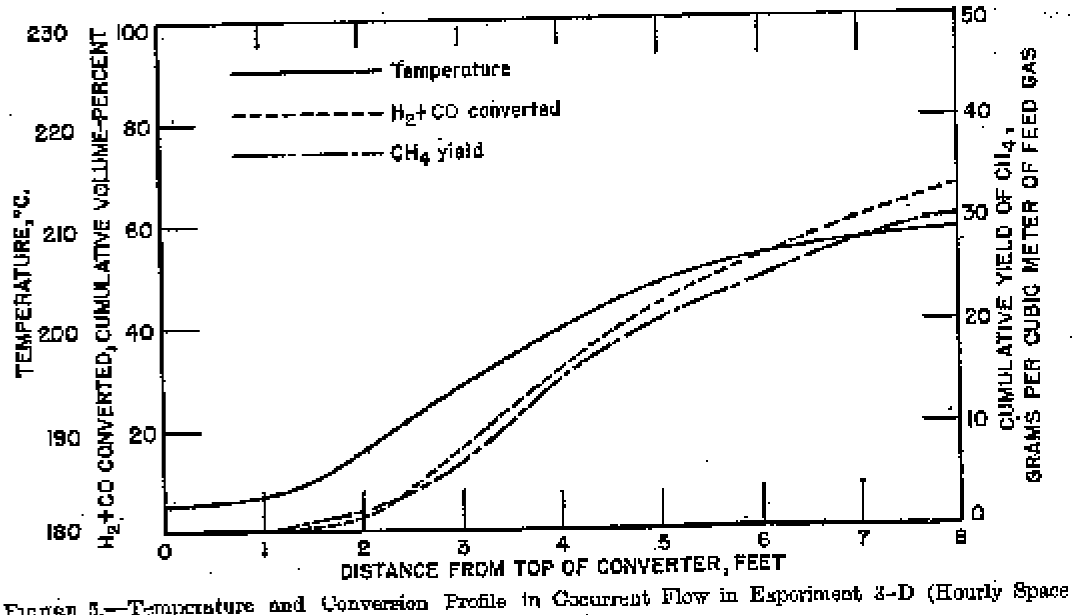

FIGURE 5.—Temperature and Conversion Profile in Cocurrent Flow in Experiment 3-D (Hourly Space Velocity, 142).

TABLE 5.—*Conversion of synthesis gas as a function of hourly space velocity with cobalt in experiment 5*

Experiment No.	5–B	5–C	5–D	5–E
Synthesis gas:				
S. V. H. (settled bed)	108	299	438	600
Conversion, vol.-percent H_2+CO	67	43	37	33
Pressure, average, p. s. i. g.	93	119	160	170
Temperature, ° C.:				
Average	184	192	201	204
Maximum	232	240	23	220
Yields:				
Specific, gm. C_3+C_4 per m.³	29.8	44.9	37.9	62.5
Space-time of C_3+, kg. per m.³ settled cat. vol. per day calculated	240	429	586	607

Although cobalt was found durable in experiments 1 to 5, further experimentation with this material was not carried out, except for a short test (experiment 6). As in the dry-bed experiments with cobalt, the liquid products were essentially completely saturated, as shown by the data of table 6. This meant production of a poor quality of gasoline with a low octane rating. The more important and motivating reasons for discontinuing the use of cobalt were concerned with its high cost, the prohibitive amounts that would be required for large-scale operation, and the potential scarcity in a time of national emergency.

TABLE 6.—*Distribution of products from cocurrent operations with cobalt*

(Weight-percent of total hydrocarbons)

Experiment No. Component	2–F (S. V. H., 102)	3–G (S. V. H., 231)
CH_4	18.5	15.9
C_2H_6	3.1	5.3
C_2H_4	.0	.0
C_3H_8	6.4	5.8
C_3H_6	1.0	.1
C_4H_{10}	7.4	7.2
C_4H_8	.7	1.8
C_5H_{12}	4.3	7.2
C_6H_{14}	4.3	5.2
C_7H_{16}	2.0	5.0
n–C_8H_{18}	3.5	4.3
n–C_9H_{20}	2.2	3.1
n–$C_{10}H_{22}$	3.3	4.0
n–$C_{11}H_{24}$	3.8	3.5
n–$C_{12}H_{26}$	2.7	3.5
n–$C_{13}H_{28}$	3.3	3.1
n–$C_{14}H_{30}$	3.3	3.1
n–$C_{15}H_{32}$	2.2	2.2
n–$C_{16}H_{34}$	3.3	2.0
n–$C_{17}H_{36}$	2.2	2.6
n–$C_{18}H_{38}$	2.1	2.2
n–$C_{19}H_{40}$	2.1	.9
n–$C_{20}H_{42}$	.5	.4
Wax	17.8	11.5
Total	100	100

As shown by the data of table 4, precipitated-iron catalysts required operating temperatures 50° to 60° C. higher than cobalt to secure comparable productivity. Higher boiling cuts of oil had to be used as coolant. Despite the increased temperatures, the specific yields of C_1 plus C_2 (25 to 35 gm. per m.³) were almost always less than those obtained with cobalt (table 3). Whereas the usage and feed-gas ratios always were similar in the cobalt tests, the usage ratio now lagged considerably. Major difficulties that had not been encountered with cobalt were disintegration and agglomeration of pelleted as well as granular precipitated iron; eventually a prohibitive pressure drop across the bed resulted.

These experiments led directly to the abandonment both of precipitated-iron catalysts and trickle-flow cooling for the remainder of the pilot-plant development. In all subsequent experiments to be discussed the submerged-bed principle was employed to effect temperature control; and the catalysts were prepared by fusion of iron oxide, except in the preliminary tests, where both pelleted cobalt and precipitated granular iron were used.

SUBMERGED, FIXED-BED EXPERIMENTS

PRELIMINARY TESTS

To determine whether temperature control could be improved by completely submerging the catalyst in the cooling medium, several submerged-bed tests were made with the same apparatus, and the same type of catalysts and cooling oil as described for the trickle-flow experiments.

The initial tests (experiments 5–F and 6) were made with pelleted cobalt. The catalyst for experiment 5–F had been used previously in trickle-flow operation, while that for experiment 6 was a fresh catalyst reduced with hydrogen. The gas saturator was bypassed during these tests, and the gas was fed directly at the bottom of the converter. The operating procedure was the same as that described for trickle-flow operation. The temperature was still controlled by varying pressure since evaporative cooling was employed. Vapors and unreacted gas left the reactor overhead and flowed to the condenser from which the condensate was returned to the reactor by a circulating pump. In experiment 5–F the liquid was returned to the top of the reactor, while in experiment 6 it was sent in at the bottom. At gas rates lower than an hourly space velocity of 300, excellent temperature control was attained; significantly, the activity of the catalyst was unimpaired. However, when this gas rate was exceeded, the gas tended to dry the catalyst at the entrance of the converter, as it had in trickle-flow operation,