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CHEMICAL PRODUCTS DIVISION

RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

A REPORT TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 7-225

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AEROJET
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RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

a report to

OFFICE OF ADVANCED RESEARCH AND TECHNOLOGY
NATIONAL AERONAUTICS AND SPACE ADMINISTRATION
WASHINGTON, D.C.

S. D. Rosenberg, G. A. Guter, F. E. Miller, and R. L. Beegle

Contract NAS 7-225

Report No. 0765-03-2 (Quarterly) January 1965

AEROJET-GENERAL CORPORATION
A SUBSIDIARY OF THE GENERAL TIRE & RUBBER COMPANY

This report is submitted in partial fulfillment of Contract NAS 7-225. The period covered by the report is 16 October 1964 through 15 January 1965.

AEROJET-GENERAL CORPORATION

S. D. Rosenberg for

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Chemical Products Division

19874

ABSTRACT

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Two successful runs were completed with the silicate reduction furnace. Both an acid basalt and a granite were reduced with methane gas; good material balances were obtained. Further modification of the methane inlet tubes is suggested. The first long duration run (60 days) with the carbon monoxide reduction apparatus was completed under a variety of conditions while maintaining satisfactory conversions and product yields. No apparent change in catalyst activity was noted.

author

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I. OBJECTIVE

The objectives of this program are (a) to study the silicate reduction process with emphasis on the design and experimental operation of a resistance-heated silicate reduction reactor, and (b) to study the effect of long-term operation of the carbon monoxide reduction reactor on catalyst life.

II. SUMMARY

The program is divided into three tasks: Task 1, Carbothermal Reduction of Silicates; Task 2, Carbon Monoxide Reduction, Methane Synthesis; and Task 3, Reports. Under Task 1, two runs of long duration were completed with the silicate reduction unit. This unit handles a charge of approximately 1 lb of silicate material (granite or acid basalt) utilizing a resistance-heated furnace. Good carbon balances were obtained in the reaction between methane and the silicate rock. Modifications of the inlet tube design are indicated for greater efficiency.

Under Task 2, the first run of long duration was completed with the carbon monoxide reduction reactor. This run was continued for 1495 hours (62 days) under a variety of conditions with excellent carbon monoxide conversion (100%) and product yields [methane (99.8%); water (98.1%)].

III. TECHNICAL DISCUSSION

A. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

1. Reactor Modifications

Heating tests were run up to 1700°C with the apparatus completely assembled and no rock in the furnace crucible.* These tests indicated that further modification of the furnace was required.

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*See Aerojet-General Quarterly Report 0765-03-1, Contract NAS 7-225 (October 1964) for a complete description of the silicate reduction unit.

a. Cooling coils were installed around the outside of the steel furnace case (see Figure 1). A test showed that these coils were sufficient to maintain the steel case below 100°C when the crucible wall temperature was 1700°C.

b. The heating coil leads were increased from 3 to 6 strands of 0.050-in. molybdenum wire and were clamped to the water-cooled copper lines extending inside of the furnace. No trouble was experienced with these leads during the 1700°C test.

c. The melting crucible was shortened from 12 in. to 8 in. (see Figure 2). The heating crucible was shortened from 14 in. to 12.5 in., and the outer shield crucible was shortened from 18 in. to 8.5 in. In addition, covers were made from zirconia (ZrO_2) for the melting crucible and for the heating crucible. These changes were made to decrease the heat losses from the top of the furnace and to lower the temperature of the gas inlet tubes.

d. A cooling coil of 1/4-in. stainless-steel tubing was installed 7 in. from the top of the furnace, and 2 in. below the top of the gas inlet bells (see Figure 2). This was done to maintain the top 2 in. of the inlet bells below the cracking temperature of methane. The empty crucible test with this coil in operation was inconclusive. The cooling coil made contact with one of the inlet bells which became electrically conductive and shorted the heating coil to ground. A new coil was made which is slightly larger in diameter. It does not make direct contact with the inlet bells. In addition, more efficient heat insulation was installed in the top part of the furnace by replacing the Al_2O_3 bubbles with ZrO_2 bubbles.

e. The inlet tubes, melting crucible, cooling coil, and furnace outlet tube were fabricated as a unit attached to the furnace cover (see Figure 2). This permits the unit to be assembled from the top. Final insulation is achieved by pouring ZrO_2 bubbles through holes in the furnace cover.

f. The following changes were made in the unit piping (see Figures 1 and 3):

(1) A rotameter and a needle valve were installed on each of the three gas inlet tube lines. This provides a means of flow control to each of the three inlets.

(2) A rotameter, needle valve, and a filter were installed in the outlet line from the inlet bells. This permits measurement and control of the flow of carbon monoxide given off from the inlet bells as the molten rock reacts with the carbon deposited therein.

(3) A filter was installed in the outlet gas line from the furnace to remove dust and/or carbon from the outlet gas stream.

(4) A 4-liter surge bottle was installed in the outlet gas lines downstream of the junction of the furnace outlet line and the outlet line from the inlet bells. This permits the two streams to mix thoroughly so that the total outlet gas composition can be accurately measured.

g. The inlet tubes and inlet bells as used in Runs 2 and 3 are shown in Figure 4. The instrument panel as modified for use in these runs is shown in Figure 5.

2. Reduction Experiments

Three silicate reduction runs were carried out this quarter. The first run was discontinued after 6 hours of heating at which time the seals around the inlet tubes started to leak. These Viton rubber seals were replaced with soft rubber seals for the second run.

a. Run 2, Reduction of Acidic Basalt

The second run was quite successful. The data for Run 2 are reported in Table 1 and Figure 6. In this run, 392 g of basalt and 8 g (2%) of carbon were charged into a 2.5-in.-dia by 8-in.-deep, 0.25-in.-wall zirconia crucible. The three inlet tubes were fabricated from alumina and mullite tubes cemented to 0.75-in.-dia by 12-in.-long zirconia bells. The bells extended to the bottom of the crucible which was filled with the basalt. The depth of the molten basalt was estimated to be about 3 in. at the start of the run; it was only about 1.5 to 2.0 in. deep at the end of the run. The crucible was placed in the furnace and heated at the rate of about 60°C/hour (see Figure 6) to 1600°C, and was then maintained at 1550 to 1600°C until most of the carbon charged (87%) was given off as carbon monoxide. An argon purge was used to protect the molybdenum furnace winding and to transport the product gas through the analytical train. Nitrogen (25°C) cooling in the stainless-steel coil was used to maintain the top several inches of the gas inlet bells below the methane cracking temperature.

After the carbon monoxide content in the product gas had dropped to below 0.5%, the methane feed gas (95.2% CH_4, 4.4% H_2, 0.4% N_2) was introduced at the rate of 0.10 g mole/hour. There was no sign of carbon dust in the exit gas stream after a few hours had elapsed, so the methane rate was increased to 0.20 g mole/hour. After this was done, there was an indication of a very small amount of carbon dust in the product gas. Methane flow was turned off after approximately seven hours of operation. The gas inlet lines had <u>not</u> clogged, and the conversion rate had not fallen appreciably when the unit was shut down (see Figure 6).

The last column in Table 1 gives the cumulative recovery, as carbon monoxide and carbon dioxide, of the carbon charged. About 87% of the carbon charged with the basalt was recovered as carbon monoxide before the methane feed gas was turned on. The overall carbon recovery was 79%, which is better than the recoveries which were achieved on the previous program when both carbon and methane were used (Reference 1).

Inspection of the apparatus after the run showed the following:

(1) The crucible remained intact throughout the run. There were no bulges, internal cracks, or serious penetration of the walls by the melt (see Figure 7).

(2) The inlet tubes (alumina surrounded by mullite) were in excellent condition. There were no cracks, bulges, or carbon deposition.

(3) The inlet bells were in fair condition (see Figure 7). One of the three inlets was shut off during the run because the seal between the inlet tube and bell opened. The other two bells operated throughout the run without failure. There was, however, an appreciable amount of carbon deposited on the inside of the bells from 5 in. to 8.5 in. above the outlet end. The unreacted carbon may be explained by assuming that the melt did not contact it [i.e., the pressure difference between the bell inlet and the outlet from the furnace was not great enough to force the melt to the top of the bell during the "vent" cycle] or the "vent" cycle (3 min out of every 30 min) was not long enough to allow the melt to react with all of the carbon that was formed during the "on" cycle. The former circumstance is much more likely to be the case.

(4) The bells cracked while cooling after the run was terminated. The inlet tubes were raised 5 in. out of the furnace over a period of 20 min after termination of methane flow in an attempt to salvage the inlet bells for another run. A slower withdrawal rate was indicated for future runs.

(5) Some of the alumina balls which were used for insulation fell into the melt when the inlet bells were withdrawn. This was the result of the crucible cover sticking to the inlet bells as they were lifted out of the furnace. In the next run, the cover was wired to the crucible, preventing a recurrence of this problem.

(6) Some trouble was experienced during the early part of the run with sublimate (a very fine white dust probably composed of sodium compounds and other low-melt materials from the basalt) clogging the product gas filter. This filter was redesigned; a second filter was installed in series with the first so the first filter can be bypassed and cleaned during a run.

(7) The product gas outlet line from the furnace (5/16-in. alumina tube) was found to be nearly clogged with sublimate. This line was increased to 3/8-in. size and brought straight up from the furnace so it could be easily cleaned during a run or could be used to add more rock to the reactor during future runs.

b. Run 3, Reduction of Granite

The data for Run 3 are reported in Table 2 and Figure 8. In this run, a charge of 454 g of granite was placed in a 2.5-in.-dia by 8-in.-deep, 0.25-in.-wall zirconia crucible. Inlet tubes similar to the ones in Run 2 were used. The crucible was placed in the furnace and heated at the rate of about 75°C/hour up to 1550°C Argon was purged through the furnace and through the inlet bells during the warmup. A very small amount of carbon monoxide (0.2%) appeared in the exit gas as the crucible temperature approached 1550°C. About 2 g of water and 0.3 g of carbon dioxide were found in the product gases up to this time. Gas samples were taken and analyzed by mass spectrophotometry (see Table 3). Samples 1 and 2, taken when the crucible was at 1150° and 1350°C, respectively, did not show any impurities such as sulfur or phosphorus compounds. The argon flow through the bells was stopped and methane feed started when the crucible temperature reached 1550°C. The methane flow was then maintained at 0.2 g mole/hour and the crucible temperature was held at 1550 to 1650°C for 15.2 hours. The run was terminated because bells 1 and 3 were becoming clogged and the conversion of methane to carbon monoxide was dropping gradually.

The rate of conversion of methane to carbon monoxide (see Table 2) increased slowly from about 40% for about 9 hours after the run was started to about 57% and then slowly declined throughout the rest of the run to 49%. Some carbon monoxide continued to be given off after the methane flow was shut off. An overall conversion of methane to carbon monoxide of 54% was obtained.

c. Methane in the Product Stream

A small amount of methane was present in the product gas stream (see Figure 8). This was due, at first, to the back flow of methane out of the bells after each cycle change. The residual methane in the bells was displaced into the product gas stream by the carbon monoxide which was generated by the rock reacting with the carbon in the bells. However, as the run progressed, the refractory cement seal between the inlet tube and inlet bells gradually opened and allowed increasing amounts of methane to bypass the bells. Part of this methane was cracked and lost as carbon deposited in the furnace; the remainder appeared in the product gas stream.

d. Carbon Balance

Table 4 presents the carbon balance obtained for Run 3. Of the 36.2 g of carbon charged as methane, only 53.6% of it was recovered as carbon monoxide and 0.6% as carbon dioxide; 7.2% was recovered as methane in the product gas. Carbon deposited in the inlet bells accounted for 20.4% of the methane charged, and carbon filtered from the outlet gas for 3.0%. The remainder, 5.5 g (15.2%), was not accounted for quantitatively; it was deposited as carbon on the insulation in the furnace. The alumina and the Fiberfrax insulation in the furnace were quite black.

e. Metal and Slag Recovered

The slag recovered from the run was a light greenish-gray glass. Microscopic examination of the slag showed that it contained many very small (less than 0.001-in. dia) nodules of metal. In addition to the microscopic nodules of metal, there were a few larger nodules of metal (up to about 1/8-in. dia). The larger ones were collected and found to weigh 0.34 g. The analysis of this metal is given in Table 5. The average of two spectrographic analyses showed that the metal was 58.3% Fe and 38.4% Si, the balance being Ni, Cu, and Ag.

f. Silica Reduction

The above results prove that some silica was reduced to silicon. However, the amount that was completely reduced was much smaller than expected. The amount of iron metal found was also small (3% of the total expected). About 1.6 g mole of carbon monoxide was recovered from the run. The potassium and sodium oxides in the granite required only 0.46 mole of carbon for reduction; the iron actually reduced required only 0.01 mole; this left 1.13 moles of carbon to reduce silica. The results indicate that suboxides of iron and silica were formed and remained in solution. Apparently, granite is much more difficult to reduce to the free metals than basalt.

g. Impurities in the Gas

A special effort was made during this run to detect any impurities or trace elements in the product gas that would poison the methanation catalyst. In order to detect any acidic gases, the product gas stream was bubbled for 6 min of each hour through a CNGA gas absorber containing 0.1 N NaOH. This liquid was then analyzed by wet methods for sulfur and phosphorus compounds (see Table 3). No sulfur was found; only 0.7 ppm of phosphorus was found. Other gas samples taken throughout the run (see Table 3) for mass spectrophotometric analyses did not contain any unexpected gases.

B. TASK 2, CARBON MONOXIDE REDUCTION, METHANE SYNTHESIS

1. Reactor Modifications

Starting with Run 1-6, another carbon monoxide feed line was added to the reactor at 12.5 in. from the bottom of the catalyst bed, thus giving five lines in all. Previously, this line had been the first of the reactor sample lines.* This modification tended to lower the overall temperature rise, thereby extending the

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*See Aerojet-General Quarterly Report 0765-03-1, Contract NAS 7-225 (October 1964) for a complete description of the methanation unit.

capacity and efficiency of the catalyst bed. Also starting with Run 1-6, a Fisher Mine-Air gas buret was installed in the Orsat gas analyzer. This long 100-ml buret is graduated in three sections from 0 to 72 ml by 0.2 ml, from 72 to 94 ml by 0.1 ml, and from 94 to 100 ml by 0.05 ml. This buret was installed to increase the accuracy of the carbon dioxide determinations; the buret previously used was graduated by 0.2 ml throughout.

2. Reduction Experiments

The first long-duration carbon monoxide reduction run was completed during this report period. The reactor was operated almost continuously for 1495 hours (62 days) with no indication of a change in performance. The data for the complete 60-day test are reported in Tables 6 through 9. Data collected under each set of reaction conditions are grouped together.

The conditions and product yields for Run 1 are listed in Tables 6 and 8, the product gas analyses are given in Table 7. The carbon monoxide conversion remained at 100% throughout the run. The percent yields of products averaged over the entire run are as follows: methane, 99.8; water 98.1; and carbon dioxide 0.4. The carbon dioxide yield became significant only toward the end of the run when a high space velocity (3000 hr^{-1}) was used. These data indicate that the activity of the catalyst remained high throughout the run, and conditions are known to obtain quantitative conversion of carbon monoxide to methane and water.

The carbon dioxide concentration at various positions along the catalyst bed are shown in Table 9. No measurable carbon monoxide was found at these sample stations. Figures 9 through 18 show the catalyst tube temperature profiles for the three mixture ratios (4:1, 3.5:1, and 3.1:1) and seven space velocities (1000, 1250, 1500, 1750, 2000, 2500, and 3000 hr^{-1}) used in Run 1. The variations are (a) relatively small increases in temperature as the space velocity is increased, (b) a small drift of the maximum temperature points up the catalyst tube, and (c) a relative deepening of the "valleys" between the maximum temperature peaks.

After Run 1-7 was completed, the hydrogen and carbon monoxide were premixed and introduced through the five feed inlets for about three hours. There was no apparent difference between the two methods of gas introduction; the carbon monoxide conversion, carbon dioxide production, and temperature profile as shown in Figure 16 remained relatively constant.

Figure 19 shows the variation in maximum catalyst bed temperature with space velocity at a constant mole ratio of 3.1:1. The rate of temperature rise decreases as the space velocity increases. At a space velocity of 3000 hr^{-1}, the maximum temperature is about 425°C with an inlet temperature of 250°C. The number in parenthesis in Figure 19 indicates the position of maximum temperature on the catalyst bed. Figure 20 indicates the rise in carbon dioxide production as the space velocity is increased, holding the mole ratio constant at 3.1:1. It appears that carbon dioxide production is the limiting factor toward increase in space velocity rather than temperature rise. Figure 21 illustrates a typical gas chromatogram for the product gas (3.1:1 mole ratio). The water vapor and carbon dioxide are removed from the gas stream by an absorption tube composed of Drierite and Ascarite before the stream enters the gas chromatograph sample loop. All of the peaks are attenuated by a factor of 2, with the exception of the methane peak which is attenuated by a factor of 100. Note the absence of carbon monoxide in the product gas.

Samples of the water produced were analyzed for higher boiling carbon-containing compounds ($>C_2$) on a Wilkens Hi Fi chromatograph with a flame detector and SE-30 column. The presence of lower boiling carbon compounds (C_1 and C_2) was checked with a conventional chromatograph with a thermistor bead detector and Carbowax 1000 column. No carbon compounds of any kind were found in the water samples analyzed by either of these methods.

IV. FUTURE WORK

A. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

Experiments on the reduction of silicates with methane will continue using modified inlet tubes. The objectives of these modifications will be to prevent the clogging of the gas inlets and to react as much of the methane carbon as possible with the rock melt to form carbon monoxide and silicon.

B. TASK 2, CARBON MONOXIDE REDUCTION SYNTHESIS

A run will be initiated at a space velocity of 1000 hr^{-1} to check the catalyst activity; the results will be compared with those obtained in Run 1-3. A 90-day run will be made at space velocities below 1000 hr^{-1}.

V. PERSONNEL

The senior staff assigned to this program was comprised of S. D. Rosenberg (Principal Investigator), G. A. Guter (Scientist), F. E. Miller, and R. L. Beegle, Jr.

VI. EXPENDITURES AND COMMITMENTS

Approximately 1080 man-hours and $14,900 were expended on Contract NAS 7-225.

REFERENCES

1. S. D. Rosenberg, G. A. Guter, and F. E. Miller, Research on Processes for Utilization of Lunar Resources, Aerojet-General Summary Report No. 2895, Contract NAS 7-225 (August 1964).

TABLE 1

ROCK REACTOR DATA FOR RUN 2*

Time	Crucible Temp (°C)	Gas Flow Rates (gram mole/hour)				Graphite and Methane Charged (g carbon)	Carbon Monoxide in Product Gas (mole %)	Carbon Recovered as CO (g carbon)	Cumulative Carbon Recovery as CO and CO_2 (mole %)
		Methane Feed	Argon Purge	N_2 Cooling	Product Gas				
11:00	850	0	1.08	12	1.10	8.00	0	0	-
12:00	930	0	1.08	12	1.16	8.00	0.7	0.10	1.2
1:00	960	0	1.08	12	1.12	8.00	1.7	0.33	4.1
2:00	1040	0	1.08	12	1.17	8.00	2.7	0.71	8.9
3:00	1060	0	1.08	12	1.15	8.00	3.3	1.12	14.0
4:00	1130	0	1.08	12	1.21	8.00	6.3	2.03	25.4
5:00	1215	0	1.08	12	1.25	8.00	4.6	2.72	34.0
6:00	1315	0	1.08	24	1.20	8.00	2.1	3.02	37.8
7:00	1350	0	1.08	24	1.21	8.00	3.1	3.47	43.4
8:00	1340	0	1.08	36	1.18	8.00	4.0	4.04	50.5
9:00	1509	0	1.08	54	1.20	8.00	4.1	4.63	57.8
10:00	1545	0	1.08	60+	1.17	8.00	6.4	5.53	69.0
11:00	1595	0	1.08	60+	1.12	8.00	4.4	6.12	76.5
12:00	1555	0	1.08	60+	1.10	8.00	3.4	6.57	82.2
1:00	1595	0	1.08	60+	1.10	8.00	1.9	6.82	85.2
2:00	1600	0	1.08	60+	1.12	8.00	0.7	6.91	86.4
3:00	1575	0	1.08	60+	1.06	8.00	0.5	6.97	87.1
4:00	1540	0.10	1.08	60+	1.25	8.57	3.6	7.51	87.5
5:00	1565	0.10	1.08	60+	1.43	9.71	7.0	8.71	89.5
6:10	1555	0.20	1.08	60+	1.54	10.70	6.6	9.93	92.6
7:10	1565	0.20	1.08	60+	1.45	12.98	5.4	10.87	83.8
8:10	1560	0.20	1.08	60+	1.45	15.26	6.2	12.05	79.1
9:10	1565	0.20	1.08	60+	1.44	16.59	6.5	13.17	79.4
10:10	1579	0.20	1.08	60+	1.48	18.89	6.5	14.32	75.8
11:10	1620	0	1.08	60+	1.24	20.32	5.4	15.13	74.5
12:00	1445	0	1.08	60+	1.22	20.32	4.0	15.71	77.4
						(Carbon in CO_2 recovered 0.33 gram)		(16.04)	(79.0)

---

* Two inlet tubes of alumina and mullite terminating in 0.75-in. OD by 12-in. long ZrO_2 bells; crucible - 2.5 in. OD by 8-in. deep impervious ZrO_2; charge - 392 g of basalt mixed with 8 g of graphite; CO_2 recovered = 1.20 g; H_2O recovered = 1.21 g; Methane feed = 95.2% CH_4, 4.4% H_2, 0.4% N_2.

TABLE 2

ROCKET REACTOR DATA FOR RUN 3*

Time	Crucible Temp (°C)	Gas Flow Rates (gram mole/hour)				Methane Charged (g carbon)	Carbon Monoxide in Product Gas (mole %)	Carbon Recovered as CO (g carbon)	Cumulative Carbon Recovered as CO (mole %)
		Methane Feed	Argon Purge	N_2 Cooling	Product Gas				
7:00	1600	0.20	1.08	60	1.64	.95	6.0	.47	50
7:30	1630	0.20	1.08	60	1.59	2.10	4.2	.87	41
8:00	1635	0.20	1.08	60	1.58	3.24	4.0	1.25	39
8:30	1640	0.20	1.08	60	1.56	4.37	5.5	1.76	40
9:00	1625	0.20	1.08	60	1.54	5.51	6.2	2.33	42
9:30	1625	0.20	1.08	60	1.53	6.65	7.0	2.98	45
10:00	1620	0.20	1.08	60	1.43	7.80	6.3	3.52	45
10:30	1620	0.20	1.08	60	1.43	8.94	6.9	4.11	46
11:00	1620	0.20	1.08	60	1.56	10.10	8.0	4.86	48
11:30	1615	0.20	1.08	60	1.58	11.21	7.9	5.61	50
12:00	1610	0.20	1.08	60	1.58	12.37	8.3	6.40	52
12:30	1640	0.20	1.08	42	1.57	13.50	8.0	7.15	53
1:00	1590	0.20	1.08	36	1.42	14.64	7.8	7.81	53
1:30	1595	0.20	1.08	36	1.62	15.80	8.0	8.59	54
2:10	1585	0.20	1.08	36	1.57	17.30	8.0	9.60	55
2:50	1605	0.20	1.08	36	1.56	18.83	7.8	10.58	56
3:30	1625	0.20	1.08	36	1.56	20.38	7.5	11.52	56
4:10	1630	0.20	1.08	36	1.54	21.86	7.1	12.40	57
4:50	1610	0.20	1.08	36	1.50	23.40	6.7	13.21	57
5:30	1615	0.20	1.08	36	1.47	25.00	6.4	13.96	56
6:10	1635	0.20	1.08	36	1.44	26.42	5.7	14.62	55
6:50	1610	0.20	1.08	36	1.43	27.98	4.8	15.17	54
7:30	1655	0.20	1.08	36	1.49	31.00	4.8	15.74	51
8:10	1625	0.20	1.08	36	1.46	32.30	4.5	16.27	50
8:50	1635	0.20	1.08	36	1.45	34.80	4.4	16.78	50
9:50	1670	0.20	1.08	36	1.43	35.80	4.1	17.48	49
10:15	1660	0	1.08	36	1.23	36.20	4.0	17.72	49
11:30	1635	0	1.08	36	1.18	36.20	3.2	18.28	50
1:00	1565	0	1.08	36	1.11	36.20	2.1	18.79	52
2:00	1560	0	1.08	36	1.08	36.20	1.1	19.03	53
4:00	1570	0	1.08	36	1.06	36.20	1.0	19.43	54

---

*Three inlet tubes of alumina and mullite terminating in 0.75-in.-OD by 12-in.-long ZrO_2 bells; crucible - 2.5-in.-OD by 8-in.-deep impervious ZrO_2; charge 454 g granite; CO_2 recovered = 0.62 g; H_2O recovered = 3.99 g; feed = 95.2% CH_4, 4.4% H_2, 0.4% N_2.

TABLE 3

ANALYSIS OF PRODUCT GAS SAMPLES FROM RUN 3 BY MASS SPECTROPHOTOMETRY*

	Sample Number				
	1	2	3	4	5
Feed Gas Flow (g mole/hour)	0	0	0.2	0.2	0.2
Temperature (°C)	1150	1350	1630	1595	1630
Argon (mole%)	98.1	84.6	81.8	68.8	68.6
O_2 (mole%)	0.2	2.6	0.2	0.2	0.1
N_2 (mole%)	1.1	12.3	0.7	0.6	1.5
H_2 (mole%)	0.2	0.3	11.1	21.9	23.3
H_2O (mole%)	0.3	0.2	0.2	-	-
CO_2 (mole%)	0.1	-	0.4	-	-
CH_4 (mole%)	-	-	0.3	0.6	1.4
CO (mole%)	-	-	5.3	7.9	5.1

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*In addition to the samples for mass spectrophotometry, a special gas sample was taken throughout the run in order to detect the presence of minor gas impurities. For six minutes of each hour, the product gas was bubbled through a gas CNGA absorption bottle containing 0.1 N NaOH. This liquid was then analyzed by wet methods for sulfur and phosphorus compounds. No sulfur (less than 1 ppm) was found; only a trace (0.7 ppm) of phosphorus was found.

TABLE 4

CARBON BALANCE FOR RUN 3

	Grams of Carbon	Percent
Carbon feed as CH_4	36.2	100.0
Carbon recovered as CO	19.4	53.6
Carbon recovered as CH_4 in outlet gas	7.2	7.2
Carbon recovered as CO_2 in outlet gas	0.2	0.6
Carbon recovered as carbon in inlet tubes	7.4	20.4
Carbon recovered as carbon in outlet filter	1.1	3.0
Estimated loss in furnace	5.5	15.2
Total	36.2	100.0

TABLE 5

ANALYSIS OF METAL RECOVERED FROM RUN 3 BY EMISSION SPECTROSCOPY

	Wt%	
Metal	Sample 1	Sample 2
Iron	58.5	58.0
Silicon	37.7	39.0
Nickel	2.1	1.8
Copper	1.3	0.8
Silver	0.4	0.4

TABLE 6

REDUCTION OF CARBON MONOXIDE WITH HYDROGEN

(Operating Data)[a]

		Reactant Flow Rate		Product Flow Rate		Pressure			Temperature	
							Catalyst Bed			
Run No.	Period of Operation (hours)	H_2 (g mole/hr)	CO (g mole/hr)	Gas (g mole/hr)	Liquid Water (g/hr)	Water Jacket (psig)	Static (psig)	ΔP (in.Hg)	Water Jacket (°C)	Catalyst Bed Maximum[b] (°C)
1-1a	26-75	3.93	0.986	1.86	-	350	76.0	0.1	220	275
1-1b	75-176	3.93	0.986	1.85	16.1	360	75.5	0.1	223	296
1-1c	176-238	3.93	0.986	1.62	16.6	355	76.0	0.1	222	299
1-2a	238-255	3.39	1.09	1.57	19.7	565	75.0	0.10	249	331
1-2b	255-279	3.39	1.09	1.67	18.2	565	75.5	0.12	248	328
1-2c	279-303	3.39	1.09	1.60	18.2	560	76.0	0.12	248	326
1-2d	303-320	3.39	1.09	1.62	18.3	575	76.0	0.11	250	327
1-2e	320-341	3.39	1.09	1.61	18.2	575	76.5	0.15	249	330
1-2f	341-386	3.39	1.09	1.60	19.1	600	77.0	0.18	253	335
1-2g	386-407	3.39	1.09	1.59	19.1	580	76.5	0.17	251	332
1-2h	407-432	3.39	1.09	1.59	19.2	570	74.5	0.13	250	331
1-2i	432-456	3.39	1.09	1.60	19.0	570	75.5	0.13	249	329
1-2j	456-475	3.39	1.09	1.52	19.4	580	75.5	0.16	250	331
1-2k	475-499	3.39	1.09	1.41	19.5	570	76.0	0.15	250	333
1-2l	499-573	3.39	1.09	1.57	19.2	580	76.0	0.16	247	330
1-3a	573-596	3.81	1.23	1.27	22.0	580	76.0	0.17	251	337
1-3b	596-619	3.81	1.23	1.27	22.1	585	76.0	0.16	252	338
1-3c	619-644	3.81	1.23	1.31	22.1	580	75.5	0.15	253	339
1-3d	644-666	3.81	1.23	1.28	22.2	580	76.0	0.15	253	339
1-3e	666-693	3.81	1.23	1.14	22.4	565	76.0	0.15	251	338
1-3f	693-714	3.81	1.23	1.31	22.5	550	76.0	0.15	249	337
1-3g	714-739	3.81	1.23	1.28	22.3	555	76.0	0.15	250	336
1-3h	739-755	3.81	1.23	1.27	22.3	580	76.0	0.15	253	337

TABLE 6 (Cont.)

		Reactant Flow Rate		Product Flow Rate		Pressure			Temperature	
							Catalyst Bed			
Run No.	Period of Operation (hours)	H_2 (g mole/hr)	CO (g mole/hr)	Gas (g mole/hr)	Liquid Water (g/hr)	Water Jacket (psig)	Static (psig)	ΔP (in.Hg)	Water Jacket (°C)	Catalyst Bed Maximum[b] (°C)
1-4a	755-778	4.77	1.54	1.62	27.2	560	76.0	0.19	249	360
1-4b	778-801	4.77	1.54	1.65	26.5	560	76.0	0.20	250	357
1-4c	801-827	4.77	1.54	1.66	27.1	560	76.0	0.18	251	358
1-4d	827-851	4.77	1.54	1.63	27.4	555	76.0	0.15	250	360
1-4e	851-875	4.77	1.54	1.66	27.4	550	76.0	0.18	249	357
1-4f	875-899	4.77	1.54	1.65	26.6	555	76.0	0.17	249	356
1-4g	899-922	4.77	1.54	1.68	27.1	570	76.0	0.16	249	356
1-4h	922-997	4.77	1.54	1.66	25.8	570	76.0	0.17	249	357
1-4i	997-1016	4.77	1.54	1.71	27.4	550	76.5	0.19	249	359
1-4j	1016-1035	4.77	1.54	1.62	26.9	540	76.5	0.19	248	357
1-5a	1035-1059	5.49	1.85	2.02	32.3	560	76.0	0.22	248	374
1-5b	1059-1081	5.49	1.85	1.27[c]	31.3	580	76.5	0.22	248	374
1-5c	1081-1106	5.72	1.85	2.02	32.1	560	76.5	0.23	249	376
1-5d	1106-1146	5.72	1.85	2.02	32.2	570	76.5	0.23	249	377
1-6a	1146-1171	6.67	2.15	2.40	37.9	565	76.0	0.25	249	376
1-6b	1171-1196	6.67	2.15	2.38	37.6	580	76.0	0.25	251	376
1-6c	1196-1215	6.67	2.15	2.38	37.7	575	76.0	0.25	250	376
1-6d	1215-1242	6.67	2.15	2.41	37.9	565	76.5	0.25	249	376
1-6e	1242-1267	6.67	2.15	2.39	37.8	570	76.5	0.25	250	376
1-6f	1267-1284	6.67	2.15	2.39	37.6	570	76.5	0.25	250	377
1-7a	1284-1308	7.62	2.46	2.74	43.6	575	76.5	0.30	251	387
1-7b	1308-1333	7.62	2.46	2.74	43.4	580	76.5	0.30	251	388
1-7c	1333-1357	7.62	2.46	2.75	43.3	575	76.5	0.30	252	391
1-7d	1357-1373	7.62	2.46	2.75	43.6	570	76.5	0.30	251	393
1-8a	1373-1396	9.53	3.07	3.59	54.2	595	76.5	0.40	252	402
1-8b	1396-1419	9.53	3.07	3.50	54.5	595	76.5	0.39	252	402
1-8c	1419-1444	9.53	3.07	3.54	54.1	580	76.5	0.39	252	403

TABLE 6 (Cont.)

Run No.	Period of Operation (hours)	Reactant Flow Rate		Product Flow Rate		Pressure			Temperature	
							Catalyst Bed			
		H_2 (g mole/hr)	CO (g mole/hr)	Gas (g mole/hr)	Liquid Water (g/hr)	Water Jacket (psig)	Static (psig)	ΔP (in.Hg)	Water Jacket (°C)	Catalyst Bed Maximum[b] (°C)
1-9a	1444-1466	11.44	3.69	4.29	63.5	570	76.0	0.50	251	409
1-9b	1466-1494	11.44	3.69	4.31	64.2	575	76.0	0.50	250	407

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[a] Using Catalyst 0765-1001-1 with CO feed equally divided among the 0, 3, 6 and 9 in. feed lines for Run No. 1-1 through 1-5, and equally divided among the 0, 3, 6, 9 and 12.5 in. feed lines for Run No. 1-6 through 1-8.

[b] The catalyst bed maximum temperature as recorded 10 in. from the bottom of the bed unless otherwise noted.

[c] Hydrogen metering orifice partially clogged: H_2/CO mole ratio = 2.97:1.

TABLE 7

PRODUCT GAS ANALYSIS

Run No.	Composition of Product Gas (vol%)				
	H_2	CO	CH_4	CO_2	H_2O
1-1a	(49.7)[a]	0.0	(49.8)[a]	0.0	(0.5)[a]
1-2a	26.2	0.0	73.2	0.0	0.5
1-2b	35.6	0.0	63.7	0.0	0.5
1-2c	35.7	0.0	61.1	0.0	0.4
1-2d	36.5	0.0	62.6	0.0	0.4
1-2e	30.2	0.0	68.8	0.0	0.4
1-2f	34.7	0.0	64.6	0.0	0.4
1-2g	34.1	0.0	65.2	0.0	0.4
1-2h	31.4	0.0	67.8	0.0	0.4
1-2i	30.2	0.0	69.0	0.0	0.4
1-2j	31.9	0.0	67.5	0.0	0.4
1-2k	31.6	0.0	67.7	0.0	0.4
1-2l	30.3	0.0	69.1	0.0	0.4
1-3a	3.5	0.0	95.8	0.1	0.4
1-3b	4.7	0.0	94.5	0.1	0.4
1-3c	3.4	0.0	95.8	0.1	0.4
1-3d	3.7	0.0	95.6	0.1	0.4
1-3e	4.0	0.0	95.2	0.1	0.4
1-3f	4.2	0.0	95.0	0.1	0.4
1-3g	2.9	0.0	96.4	0.1	0.4
1-3h	2.2	0.0	97.0	0.1	0.4
1-4a	6.7	0.0	94.8	0.05	0.3
1-4b	7.6	0.0	91.7	0.05	0.3
1-4c	7.6	0.0	91.7	0.05	0.3
1-4d	5.2	0.0	94.1	0.05	0.3
1-4e	6.0	0.0	93.2	0.05	0.3
1-4f	5.3	0.0	94.0	0.05	0.3
1-4g	5.9	0.0	93.4	0.05	0.3
1-4h	9.0	0.0	90.2	0.05	0.3
1-4i	11.4	0.0	87.8	0.05	0.3
1-4j	5.6	0.0	93.4	0.05	0.3
1-5a	6.1	0.0	91.6	1.0[b]	0.3
1-5b	6.2	0.0	92.0	1.3[b]	0.3
1-5c	8.0	0.0	91.1	0.3	0.3
1-5d	7.8	0.0	91.25	0.25	0.3

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[a] Average value for this portion of the run.

[b] Hydrogen metering orifice partially clogged: H_2/CO mole ratio = 2.97:1.

TABLE 7 (cont.)

Run No.	Composition of Product Gas (vol%) H_2	CO	CH_4	CO_2	H_2O
1-6a	8.5	0.0	90.6	0.35	0.3
1-6b	8.1	0.0	90.8	0.35	0.3
1-6c	8.4	0.0	90.5	0.30	0.3
1-6d	8.2	0.0	90.8	0.25	0.3
1-6e	7.8	0.0	91.1	0.30	0.3
1-6f	7.9	0.0	90.9	0.35	0.3
1-7a	7.9	0.0	91.2	0.35	0.3
1-7b	7.7	0.0	91.4	0.35	0.3
1-7c	7.6	0.0	91.4	0.45	0.3
1-7d	8.1	0.0	91.0	0.35	0.3
1-8a	12.2	0.0	87.0	0.35	0.3
1-8b	12.1	0.0	86.9	0.35	0.3
1-8c	12.5	0.0	86.5	0.45	0.3
1-9a	10.2	0.0	86.3	1.55	0.3
1-9b	9.6	0.0	86.2	1.45	0.3

TABLE 8

CARBON MONOXIDE CONVERSIONS AND PRODUCT YIELDS

Run No.	Nominal H_2/CO Mole Ratio	Space Velocity (hr^{-1})	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		
					H_2O	CH_4	CO_2
1-1a	4:1	1000	220	100	-	-	0.00
1-1b	4:1	1000	220	100	92.5	98.6	0.00
1-1c	4:1	1000	220	100	94.8	100.5	0.00
				average	93.4	99.6	0.00
1-2a	3.5:1	1000	250	100	101.5	105.6	0.00
1-2b	3.5:1	1000	250	100	94.4	97.8	0.00
1-2c	3.5:1	1000	250	100	94.3	89.8	0.00
1-2d	3.5:1	1000	250	100	94.3	93.3	0.00
1-2e	3.5:1	1000	250	100	94.3	102.2	0.00
1-2f	3.5:1	1000	250	100	98.5	95.4	0.00
1-2g	3.5:1	1000	250	100	98.5	95.6	0.00
1-2h	3.5:1	1000	250	100	94.2	99.5	0.00
1-2i	3.5:1	1000	250	100	97.6	101.8	0.00
1-2j	3.5:1	1000	250	100	100.1	94.7	0.00
1-2k	3.5:1	1000	250	100	100.8	87.7	0.00
1-2l	3.5:1	1000	250	100	99.0	99.7	0.00
				average	97.2	97.0	0.00
1-3a	3.1:1	1000	250	100	98.4	99.2	0.1
1-3b	3.1:1	1000	250	100	98.9	97.9	0.1
1-3c	3.1:1	1000	250	100	98.9	102.7	0.1
1-3d	3.1:1	1000	250	100	99.2	99.7	0.1
1-3e	3.1:1	1000	250	100	100.1	88.8	0.1
1-3f	3.1:1	1000	250	100	100.6	101.9	0.1
1-3g	3.1:1	1000	250	100	99.7	100.5	0.1
1-3h	3.1:1	1000	250	100	99.7	100.5	0.1
				average	99.4	99.5	0.1
1-4a	3.1:1	1250	250	100	100.2	100.3	0.1
1-4b	3.1:1	1250	250	100	97.7	98.7	0.1
1-4c	3.1:1	1250	250	100	99.9	99.3	0.1
1-4d	3.1:1	1250	250	100	101.0	100.1	0.1
1-4e	3.1:1	1250	250	100	101.0	100.9	0.1
1-4f	3.1:1	1250	250	100	98.0	101.2	0.1
1-4g	3.1:1	1250	250	100	99.9	102.3	0.1
1-4h	3.1:1	1250	250	100	95.1	97.8	0.1
1-4i	3.1:1	1250	250	100	101.0	97.9	0.1
1-4j	3.1:1	1250	250	100	99.2	98.8	0.1
				average	99.6	99.7	0.1

TABLE 8 (Cont.)

Run No.	Nominal H_2/CO Mole Ratio	Space Velocity (hr^{-1})	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		
					H_2O	CH_4	CO_2
1-5a	3.1:1	1500	250	100	98.9[a]	101.6[a]	1.1[a]
1-5b	3.1:1	1500	250	100	95.9[a]	-[a]	1.6[a]
1-5c	3.1:1	1500	250	100	96.7	99.2	0.3
1-5d	3.1:1	1500	250	100	97.4	96.2	0.2
				average	97.1	97.7	0.3
1-6a	3.1:1	1750	250	100	98.0	101.7	0.3
1-6b	3.1:1	1750	250	100	97.1	100.5	0.3
1-6c	3.1:1	1750	250	100	98.1	101.1	0.3
1-6d	3.1:1	1750	250	100	98.2	102.4	0.2
1-6e	3.1:1	1750	250	100	97.6	101.5	0.3
1-6f	3.1:1	1750	250	100	98.1	102.7	0.3
				average	97.9	101.7	0.3
1-7a	3.1:1	2000	250	100	98.6	102.2	0.3
1-7b	3.1:1	2000	250	100	98.3	102.4	0.3
1-7c	3.1:1	2000	250	100	97.7	102.4	0.4
1-7d	3.1:1	2000	250	100	99.4	102.9	0.3
				average	98.5	102.5	0.3
1-8a	3.1:1	2500	250	100	97.8	100.5	0.4
1-8b	3.1:1	2500	250	100	99.0	101.7	0.4
1-8c	3.1:1	2500	250	100	97.9	100.6	0.5
				average	98.2	100.9	0.4
1-9a	3.1:1	3000	250	100	96.1	99.8	1.7
1-9b	3.1:1	3000	250	100	97.1	101.7	1.7
				average	96.6	100.8	1.7
				Run 1 average	98.1	99.8	0.4

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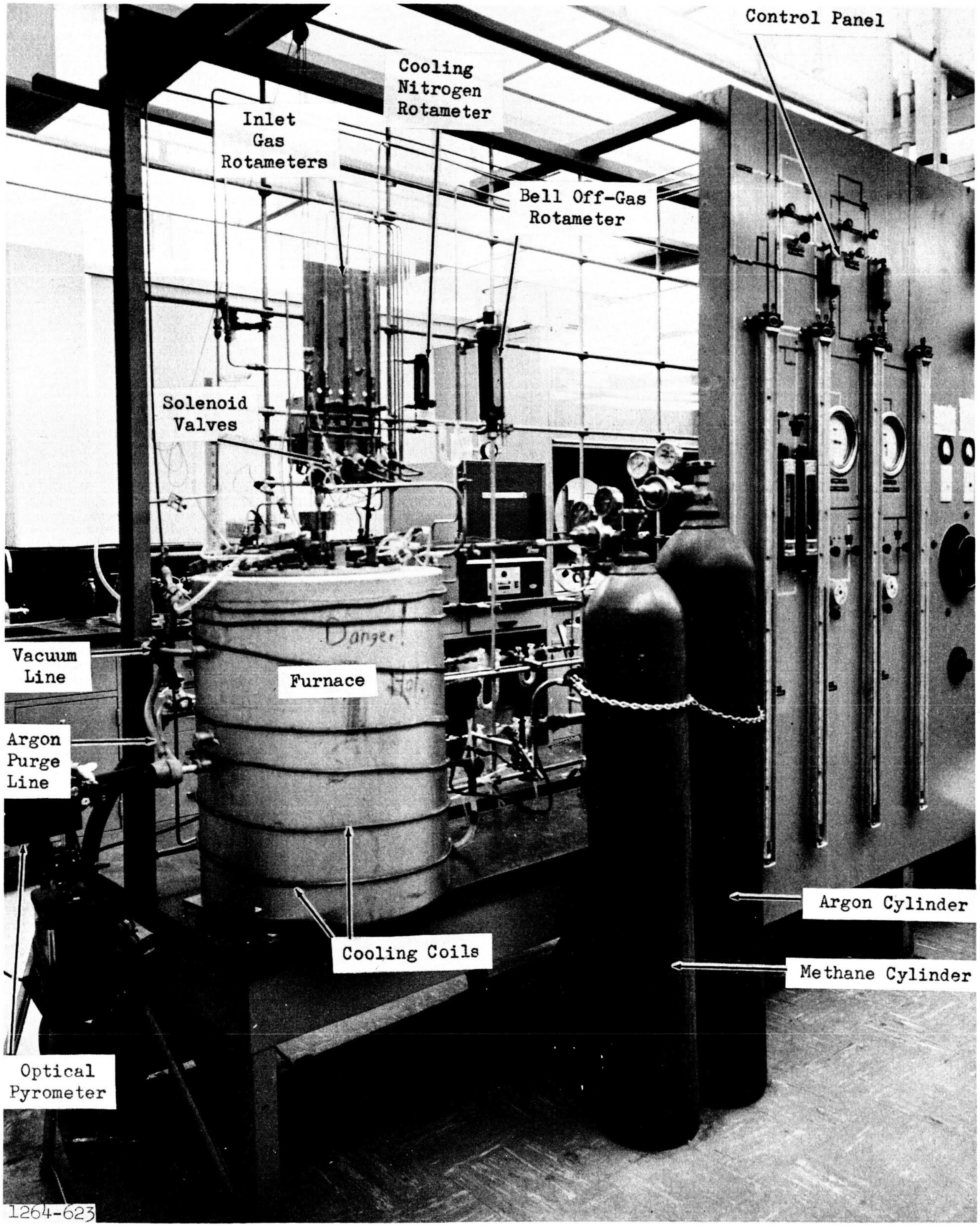
[a] Discounting 1-5a and 1-5b

TABLE 9

COLUMN SURVEY FOR CARBON DIOXIDE

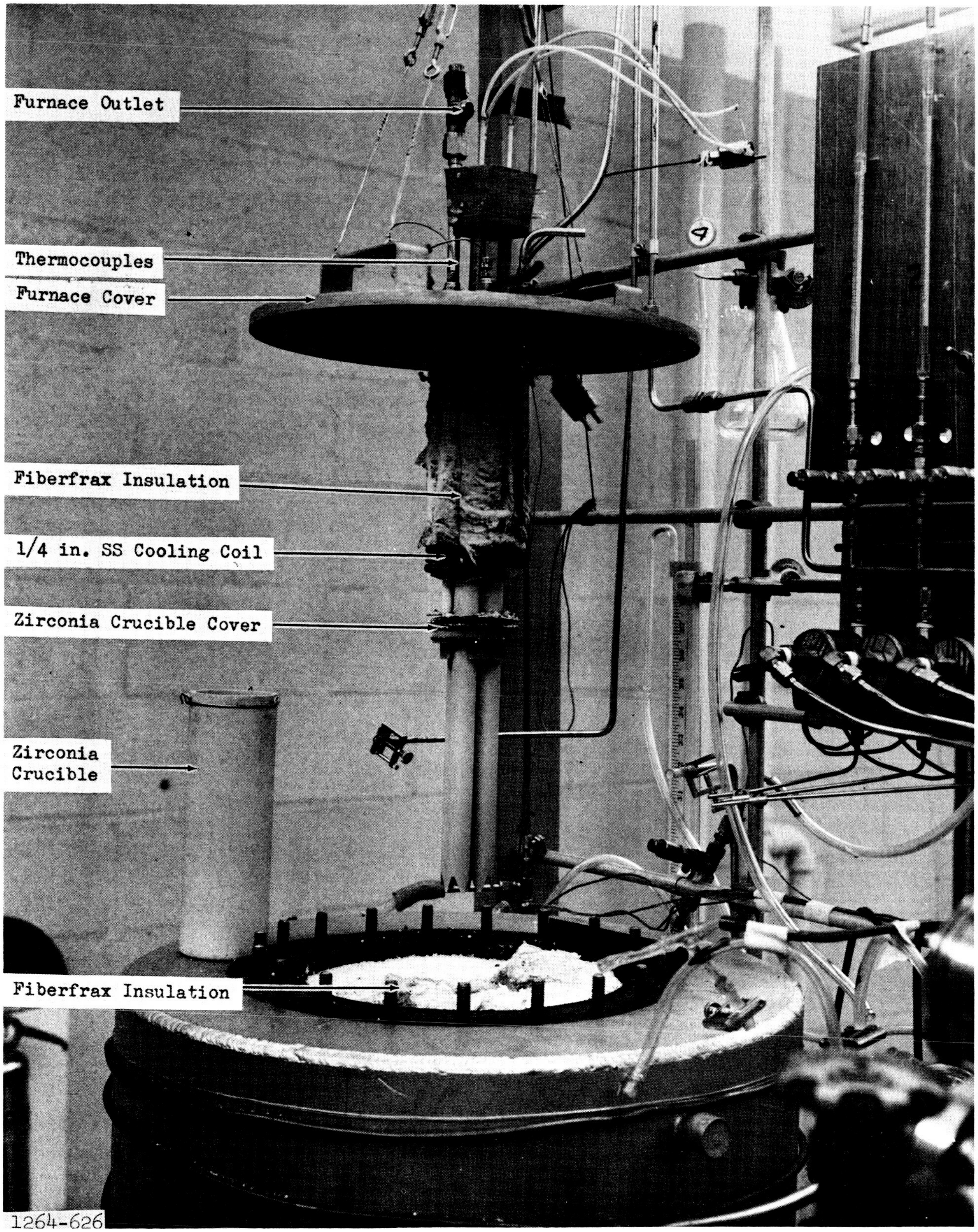
Run No.	Space Velocity (hr^{-1})	Nominal H_2/CO Mole Ratio	CO_2 Concentration (mole %) in Reactant Gas Sampled From Several Catalyst Bed Positions (in.)			
			12.5	19.0	25.5	32.0
1-1	1000	4.0:1	0.6	0.0	0.0	0.0
1-2	1000	3.5:1	1.0	0.2	0.0	0.0
1-3	1000	3.1:1	1.6	0.6	0.2	0.0
1-4	1250	3.1:1	-	1.1	0.1	0.1
1-6	1750	3.1:1		2.5	0.9	0.2
1-7	2000	3.1:1		4.0	1.5	0.8
1-8	2500	3.1:1		4.4	1.8	1.0
1-9	3000	3.1:1		4.8	2.7	1.8

Note: In Run Nos. 1-6 through 1-9, the 12.5 in. sample line was used as the fifth CO inlet.



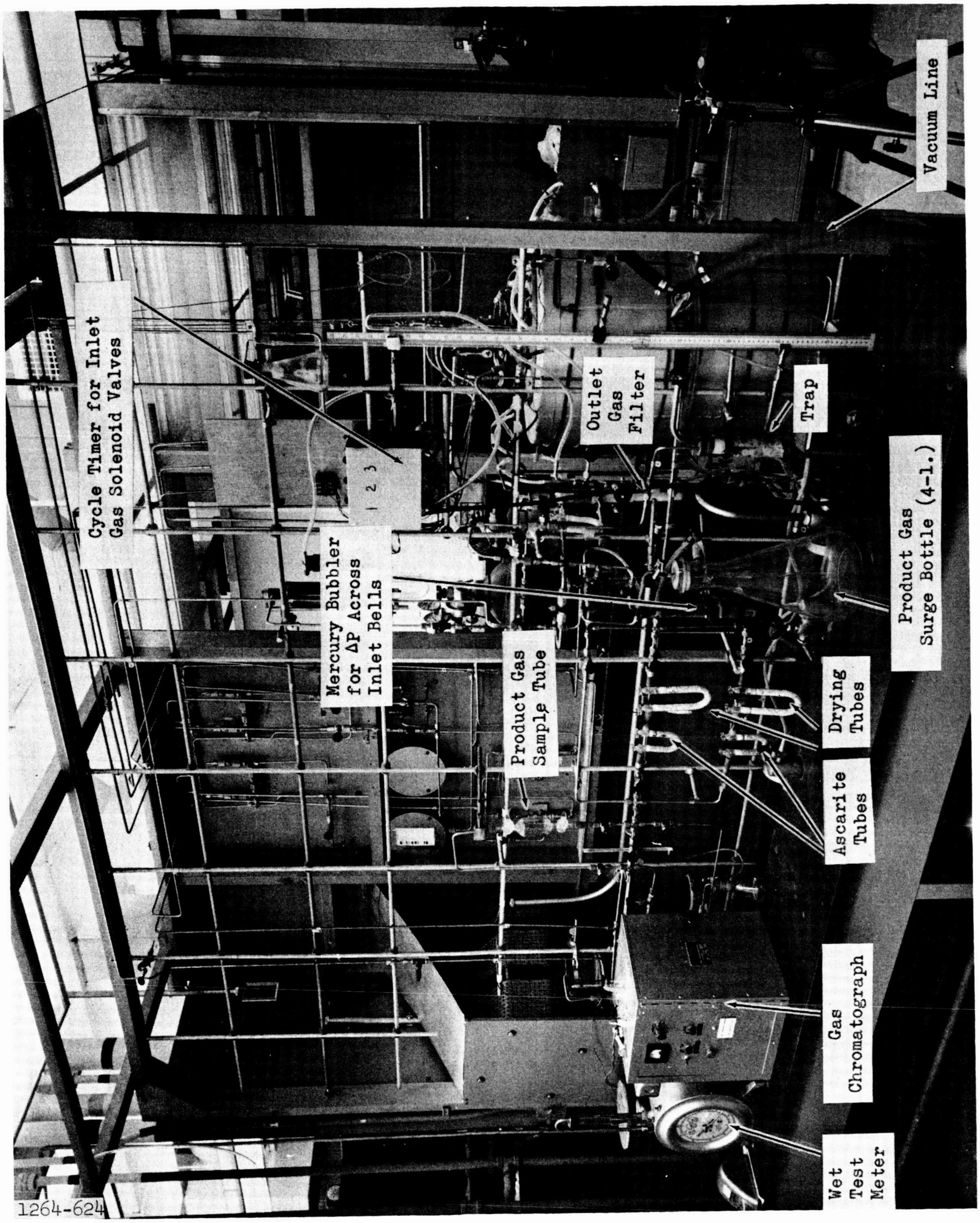
Silicate Rock Reduction Furnace Assembly

Figure 1



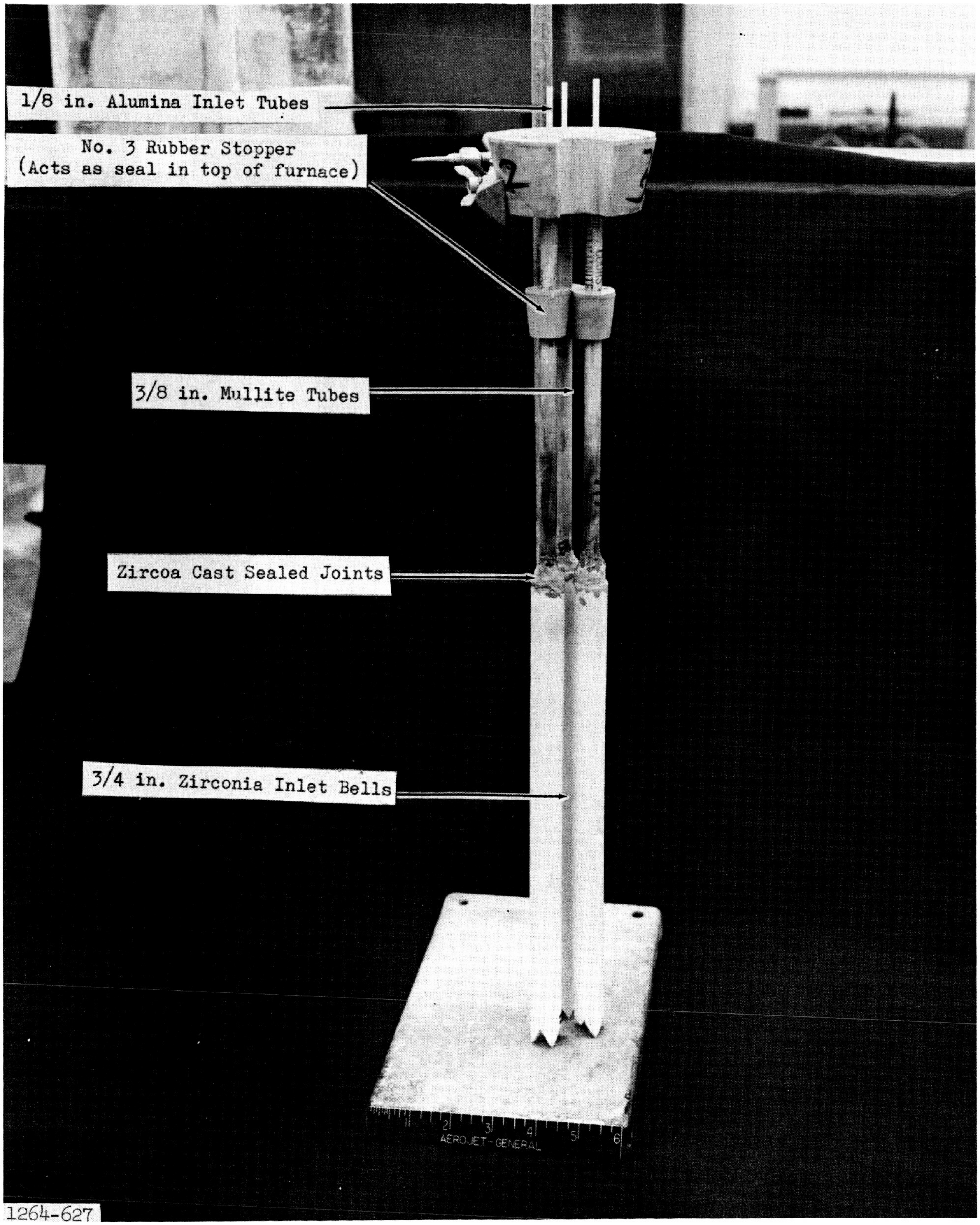
Furnace Top Ready for Assembly

Figure 2



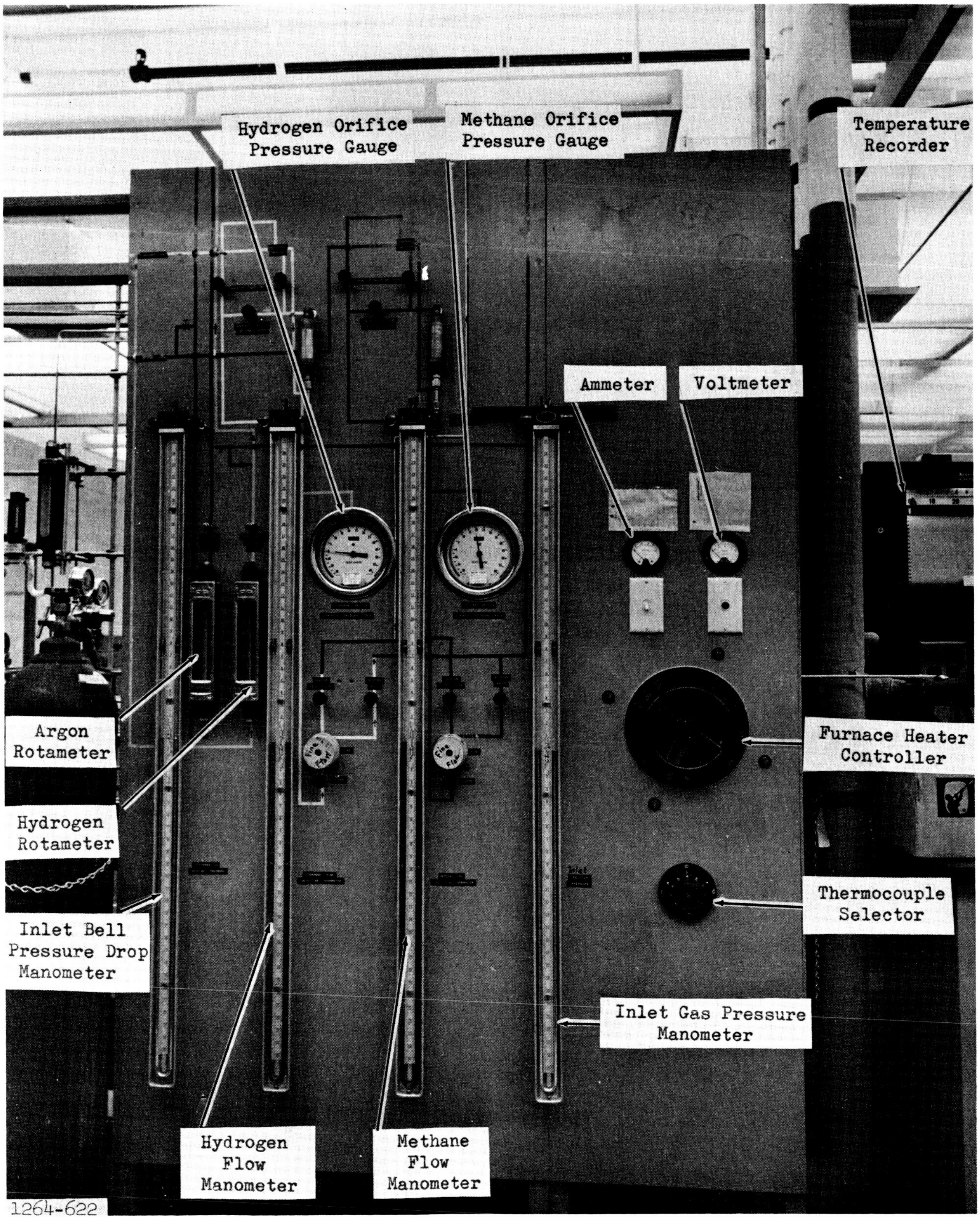
Product Gas Manifold, Rock Reduction Furnace

Figure 3



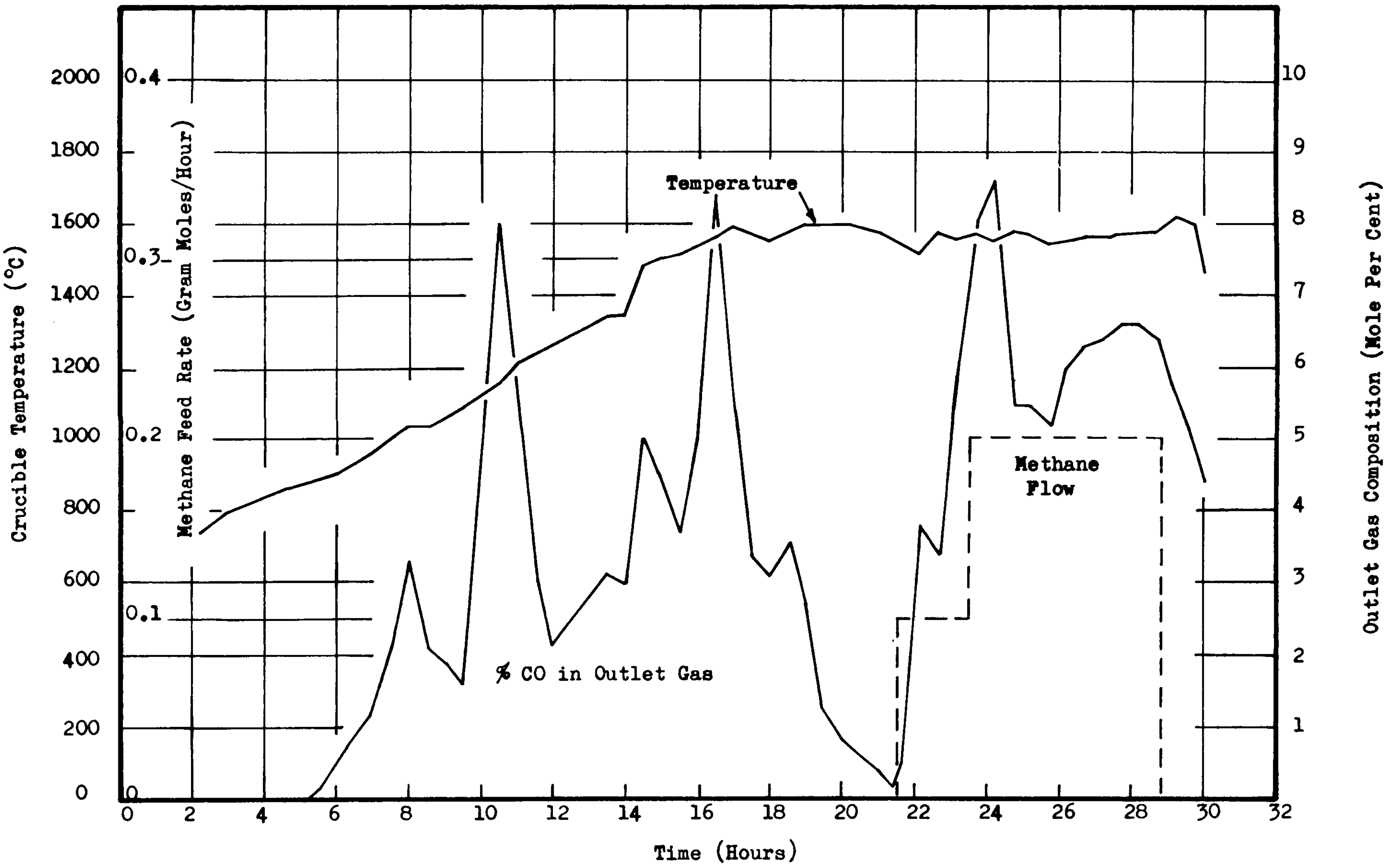
Reactant Gas Inlets

Figure 4



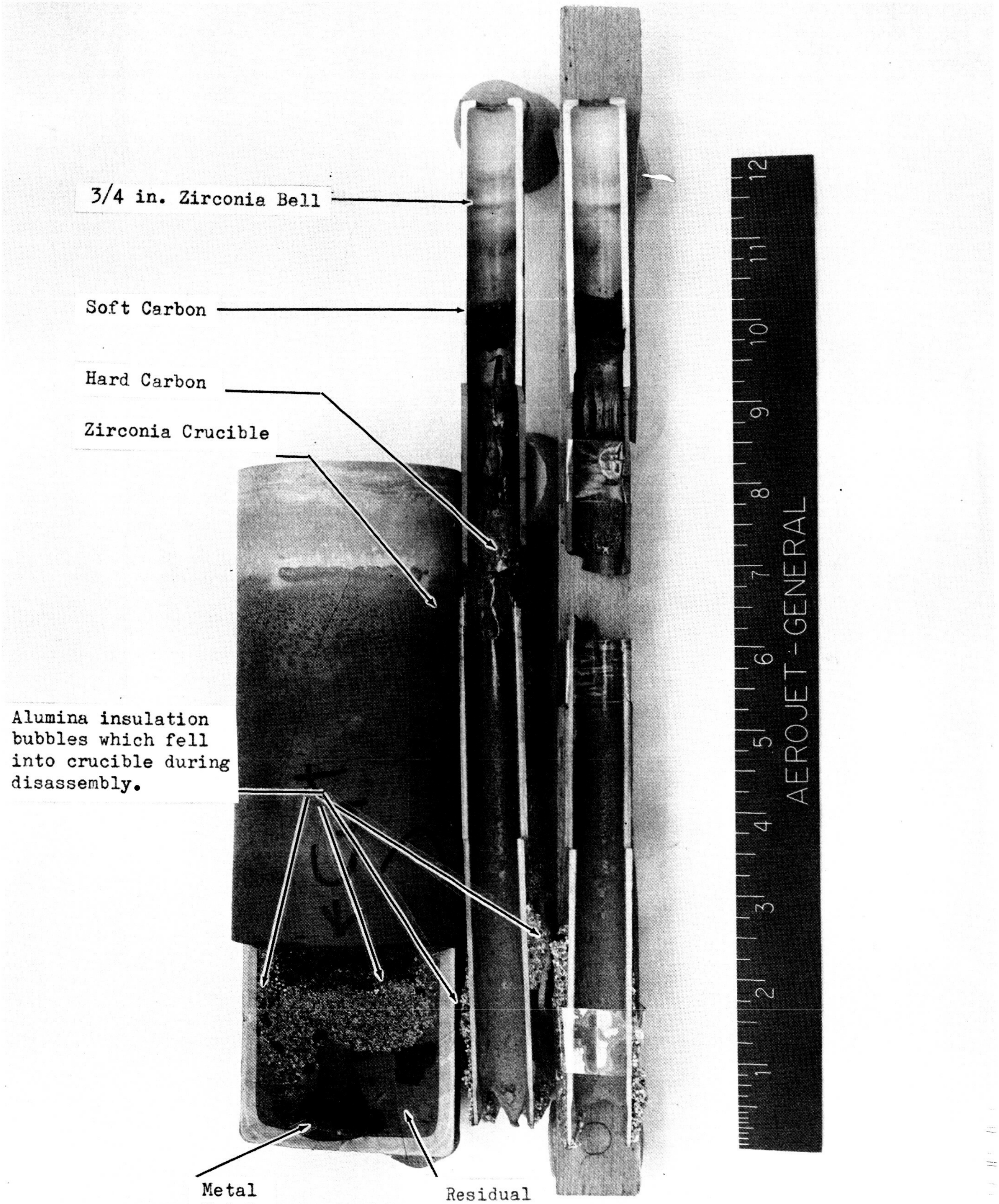
Control Panel, Rock Reduction Furnace

Figure 5



Log of Run 2, Reduction of Basalt with Carbon and Methane

Figure 6



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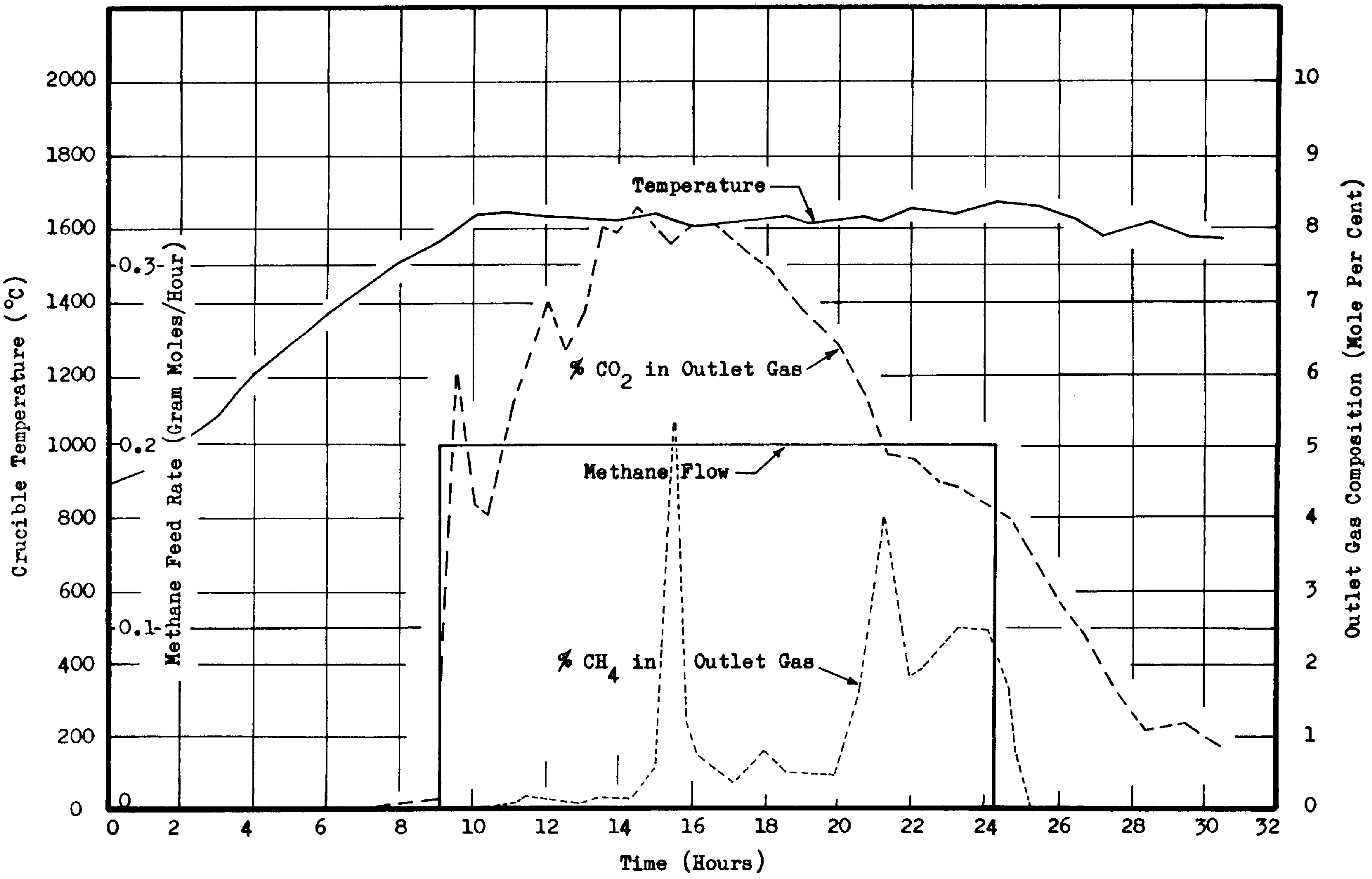
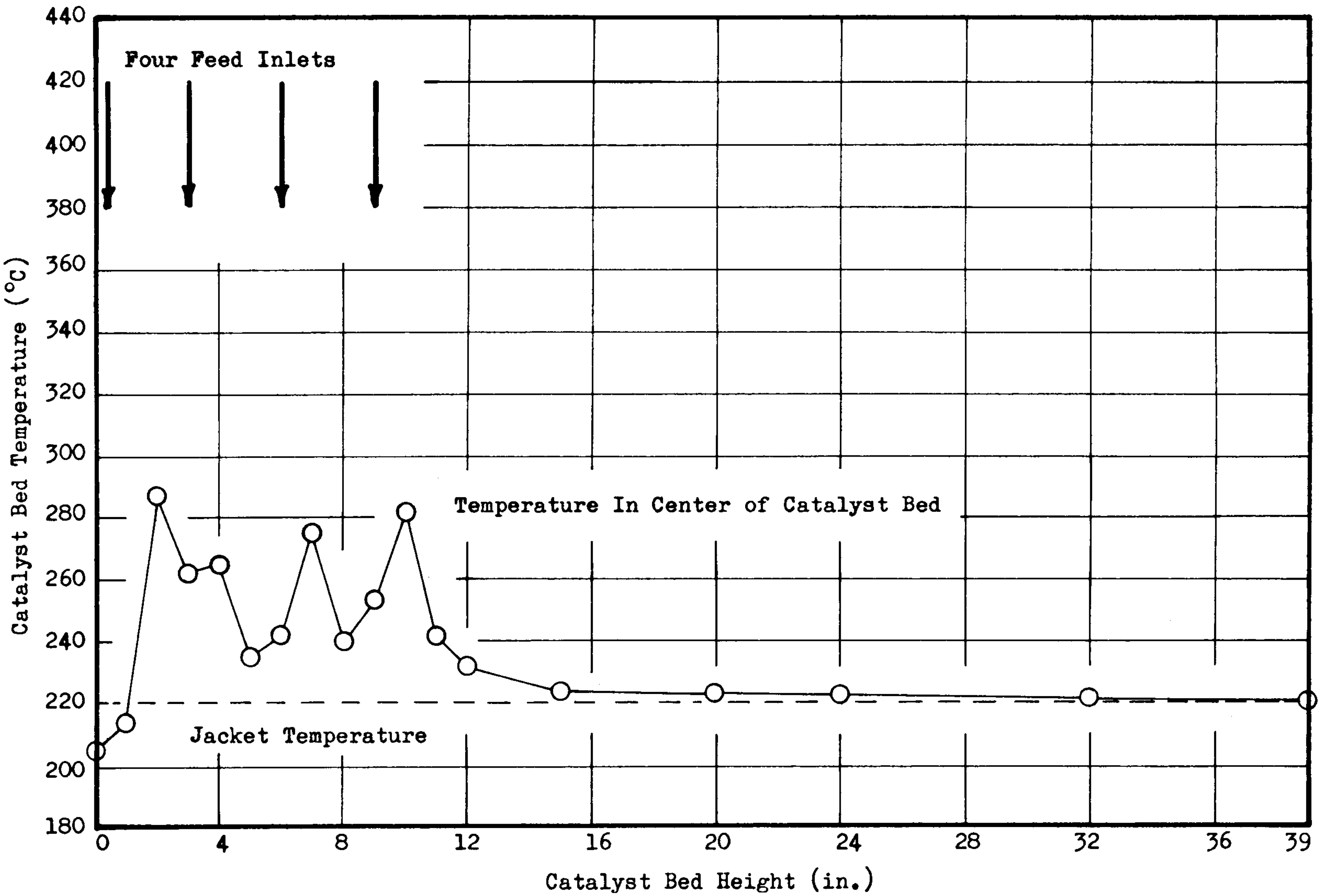
Zirconia Crucible and Inlet Bells from Run 2

Figure 7



Log of Run 3, Reduction of Granite with Methane

Figure 8

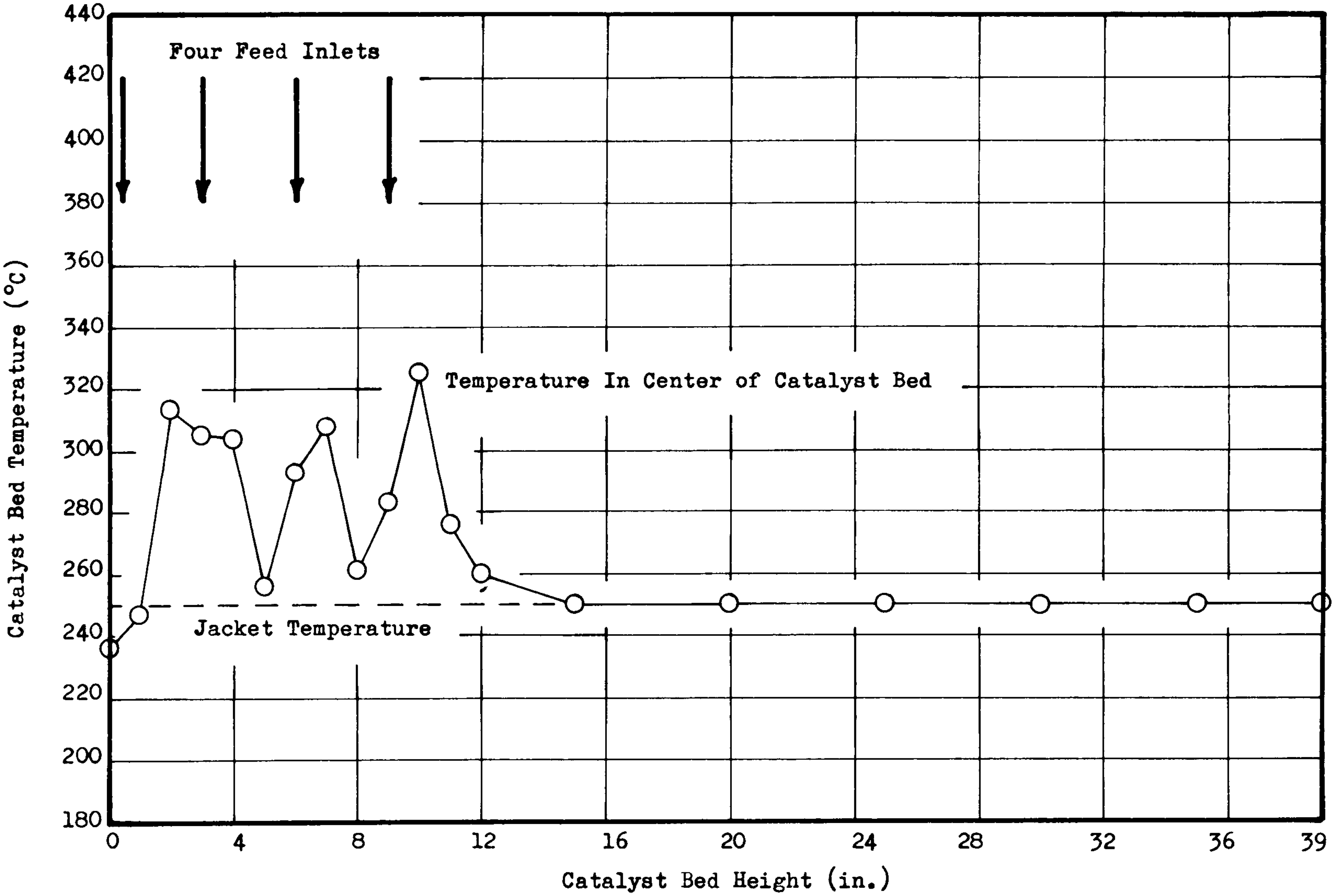


Date = 9-29-4, H_2/CO Mole Ratio = 4.0, Pressure = 6.0 atm, Space Velocity = 1000 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-25%, 3"-25%
6"-25%, 9"-25%

Catalyst Bed Temperature Profile After 30 Hours Operation

Figure 9

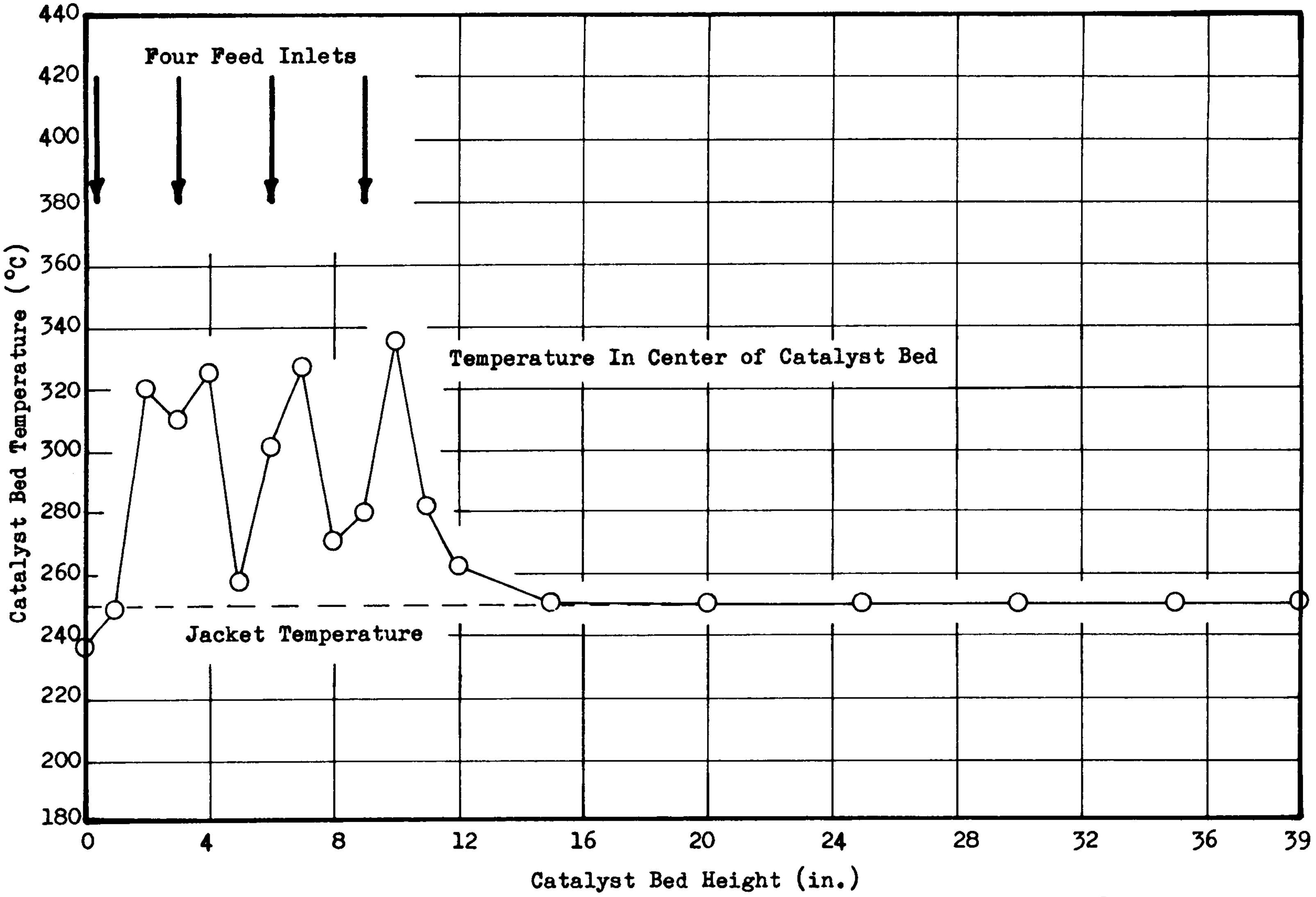


Date = 10-20-4, H_2/CO Mole Ratio = 3.5, Pressure = 6.0 atm, Space Velocity = 1000 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-25%, 3"-25%
6"-25%, 9"-25%

Catalyst Bed Temperature Profile After 574 Hours Operation

Figure 10

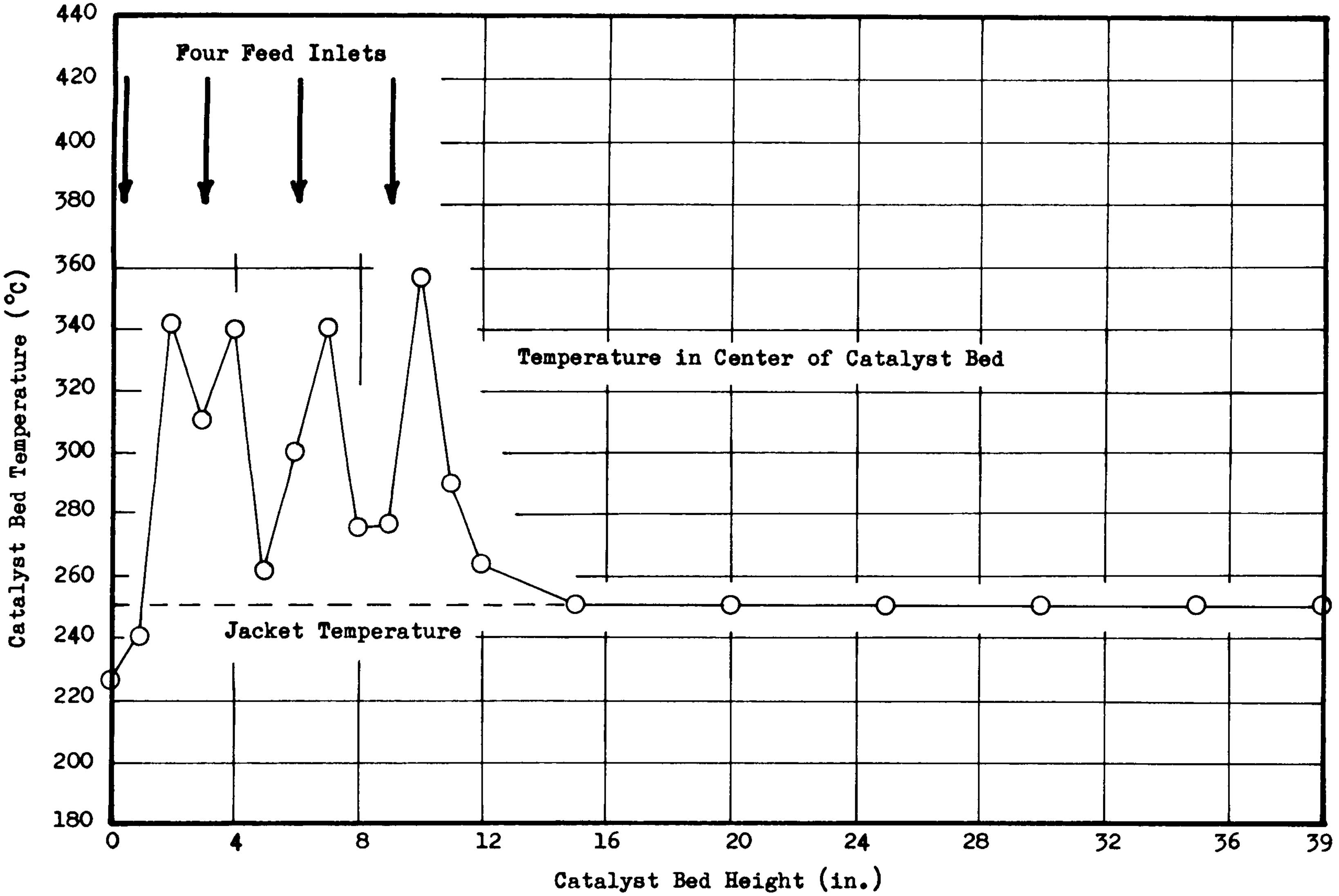


Date = 10-28-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 1000 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-25%, 3"-25%
6"-25%, 9"-25%

Catalyst Bed Temperature Profile After 757 Hours Operation

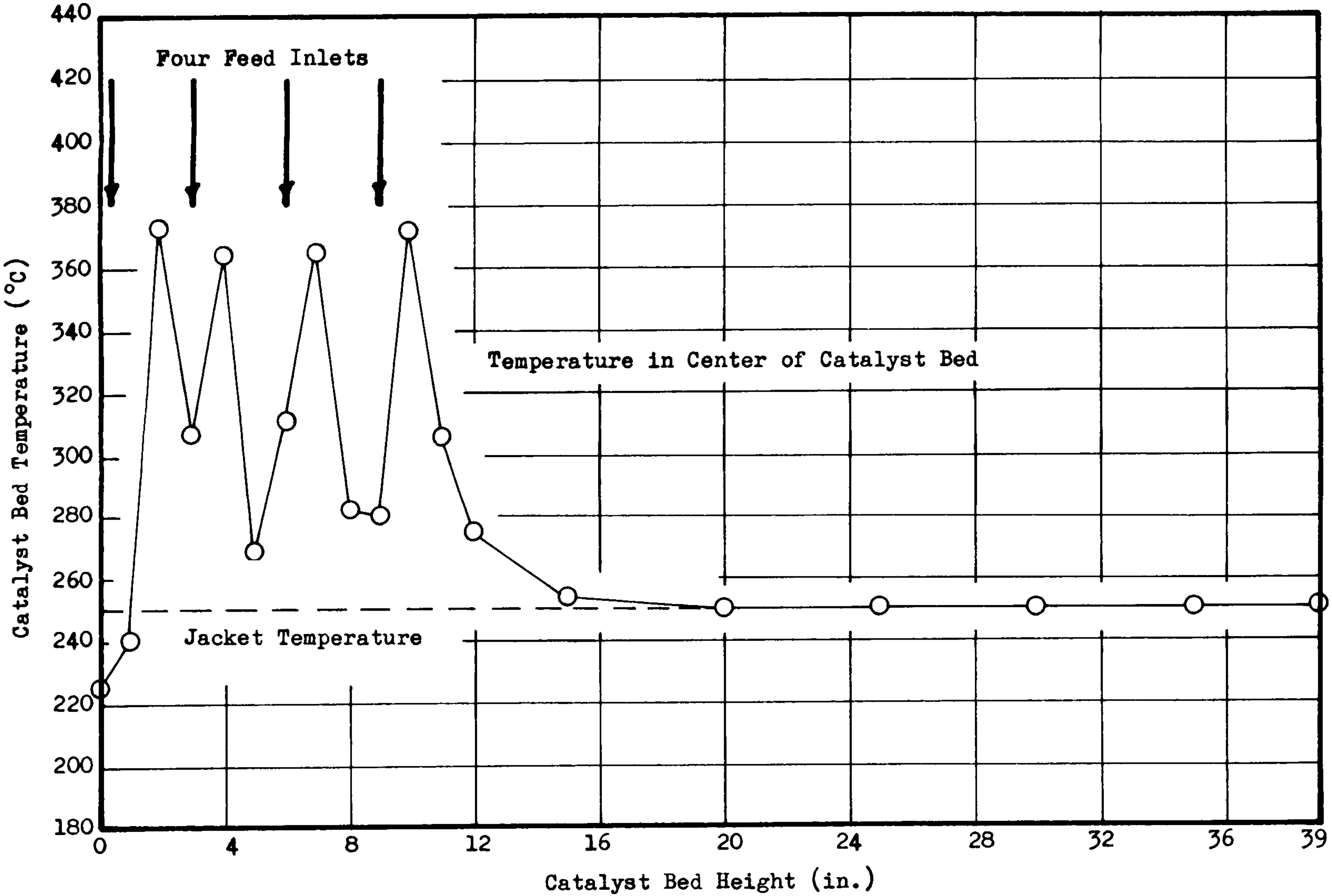
Figure 11



Date = 11-11-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 1250 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-25%, 3"-25%
6"-25%, 9"-25%

Catalyst Bed Temperature Profile After 1038 Hours Operation

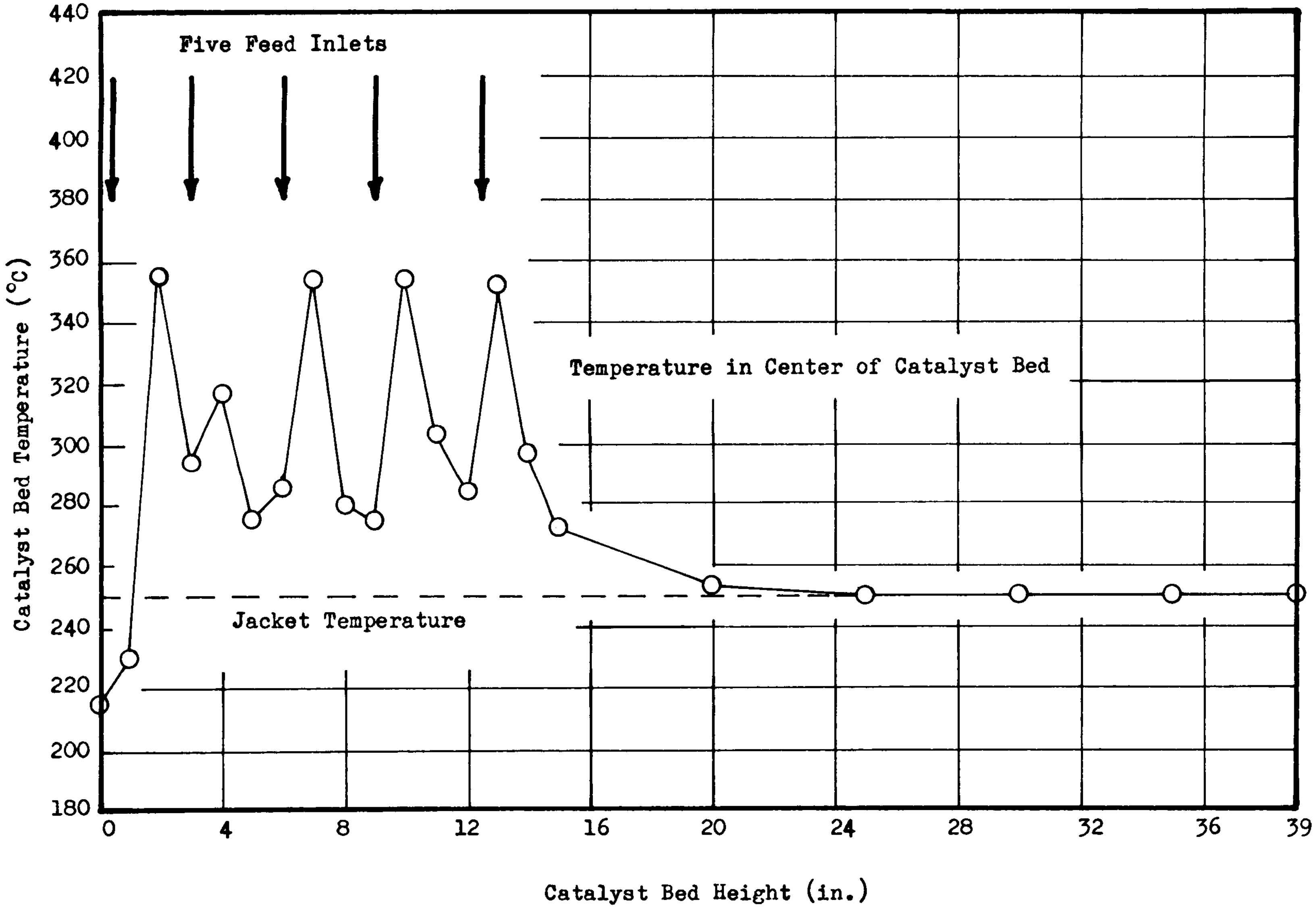


Date = 11-12-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 1500 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-25%, 3"-25%
6"-25%, 9"-25%

Catalyst Bed Temperature Profile After 1055 Hours Operation

Figure 13

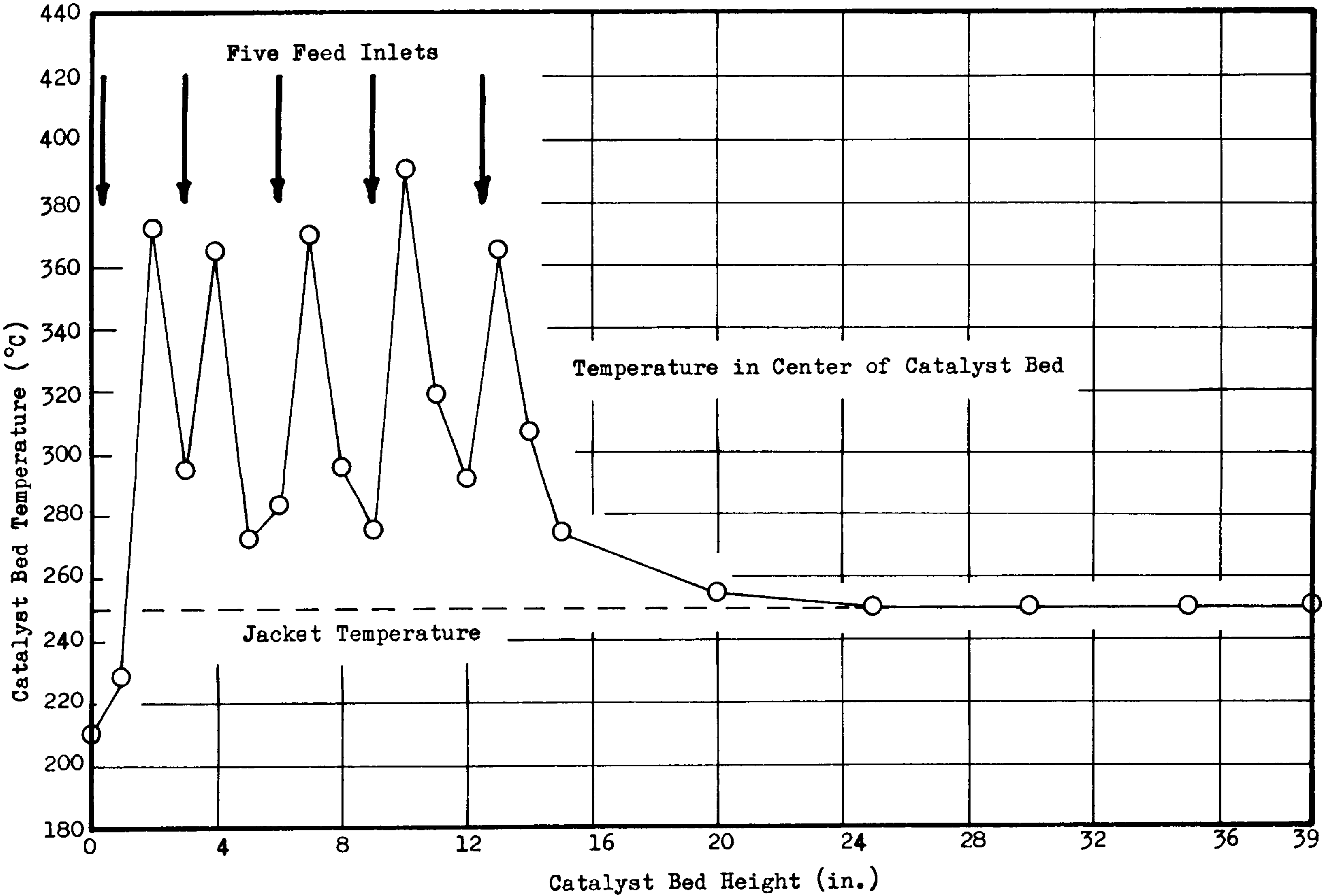


Date = 11-25-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 1750 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-20%, 3"-20%, 6"-20%
9"-20%. 12.5"-20%

Catalyst Bed Temperature Profile After 1285 Hours Operation

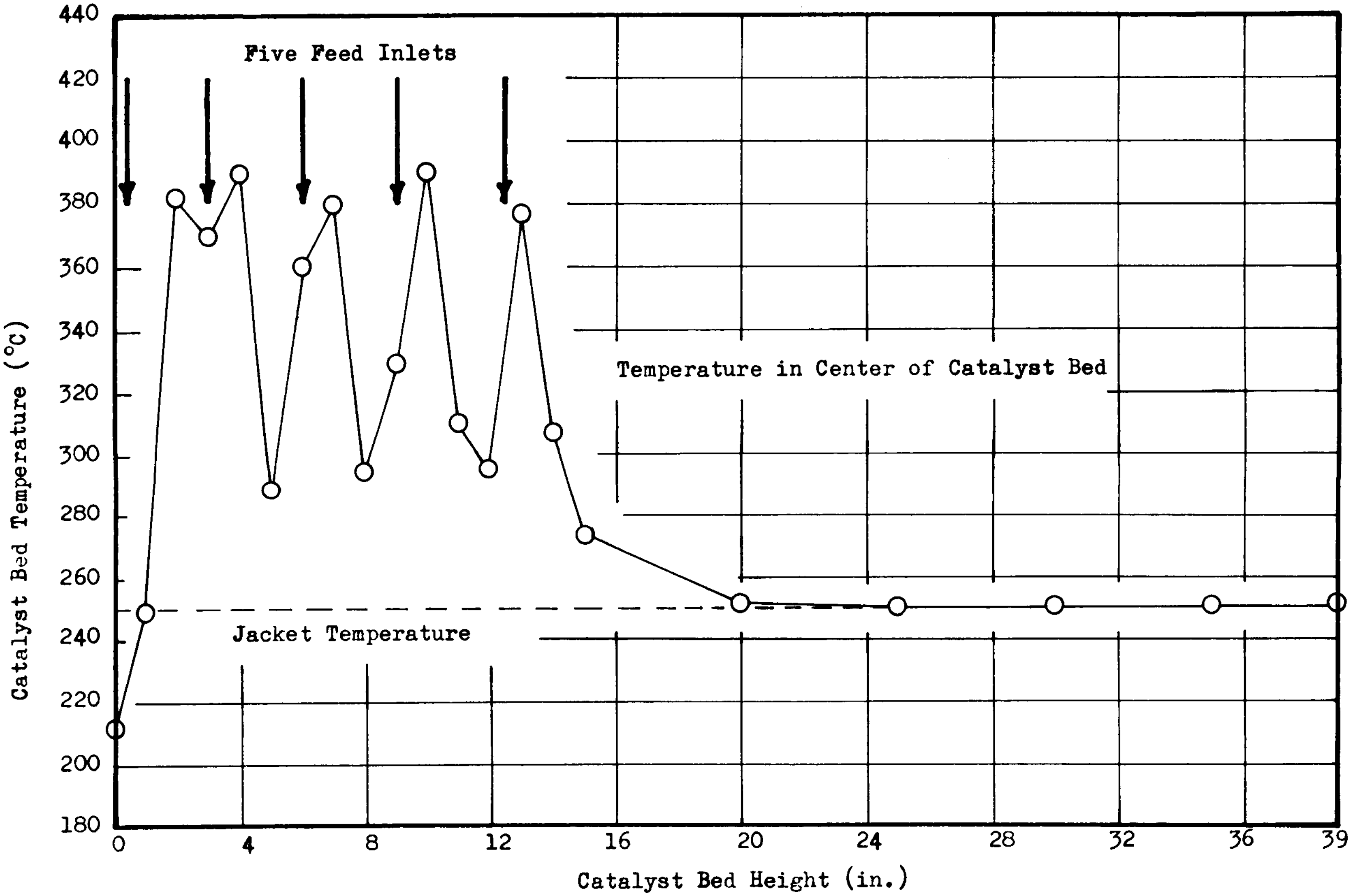
Figure 14



Date = 12-4-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 2000 hr^{-1}, Catalyst 0765-1001-1.

Feed Distribution: 0"-20%, 3"-20%, 6"-20%
9"-20%, 12.5"-20%

Catalyst Bed Temperature Profile After 1373 Hours Operation

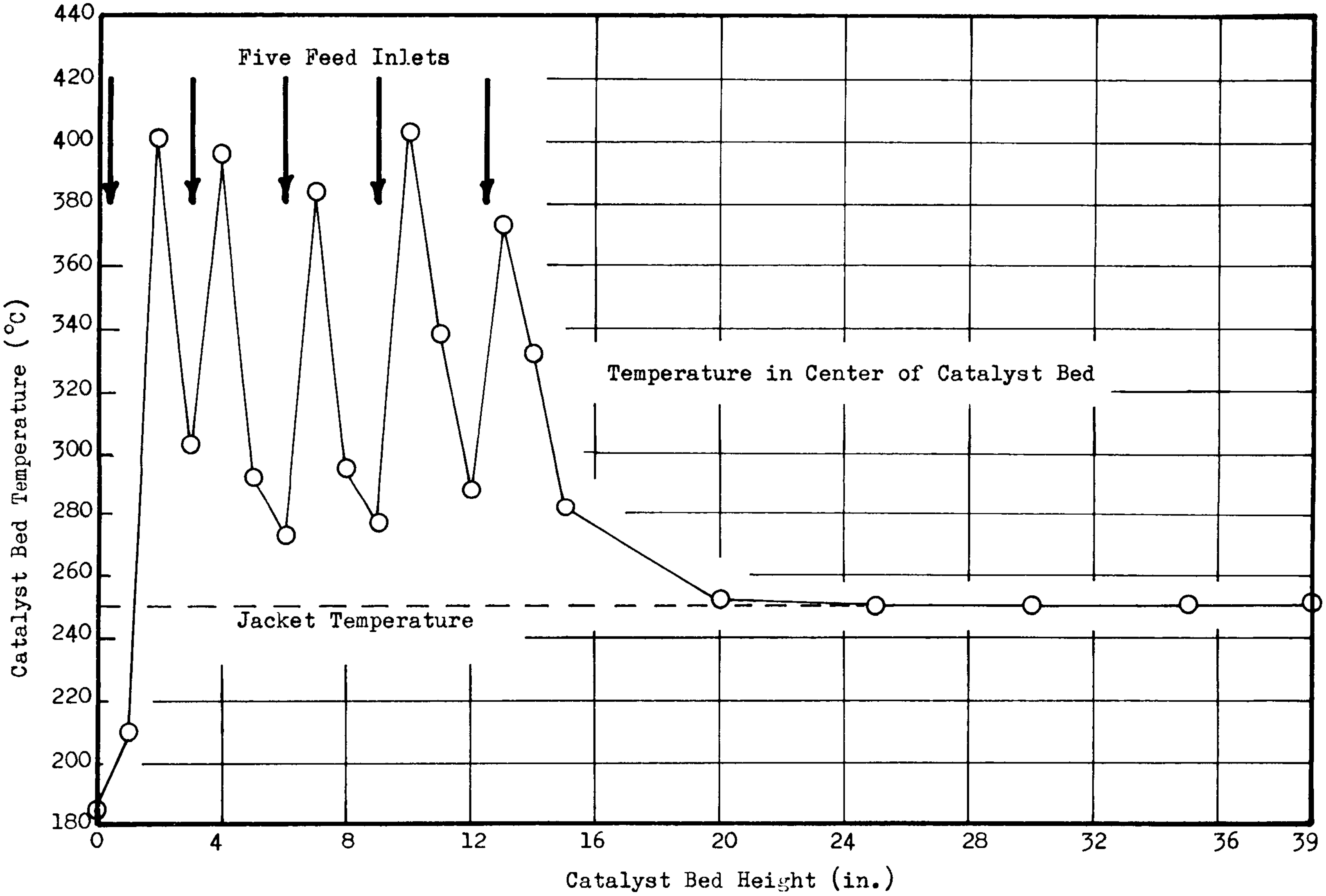


Date = 12-4-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 2000 hr^{-1}, Catalyst 0765-1001-1

Feed Distribution (H_2 and CO premixed): 0"-20%, 3"-20%, 6"-20%
9"-20%, 12.5"-20%

Catalyst Bed Temperature Profile After 1375 Hours Operation

Figure 16

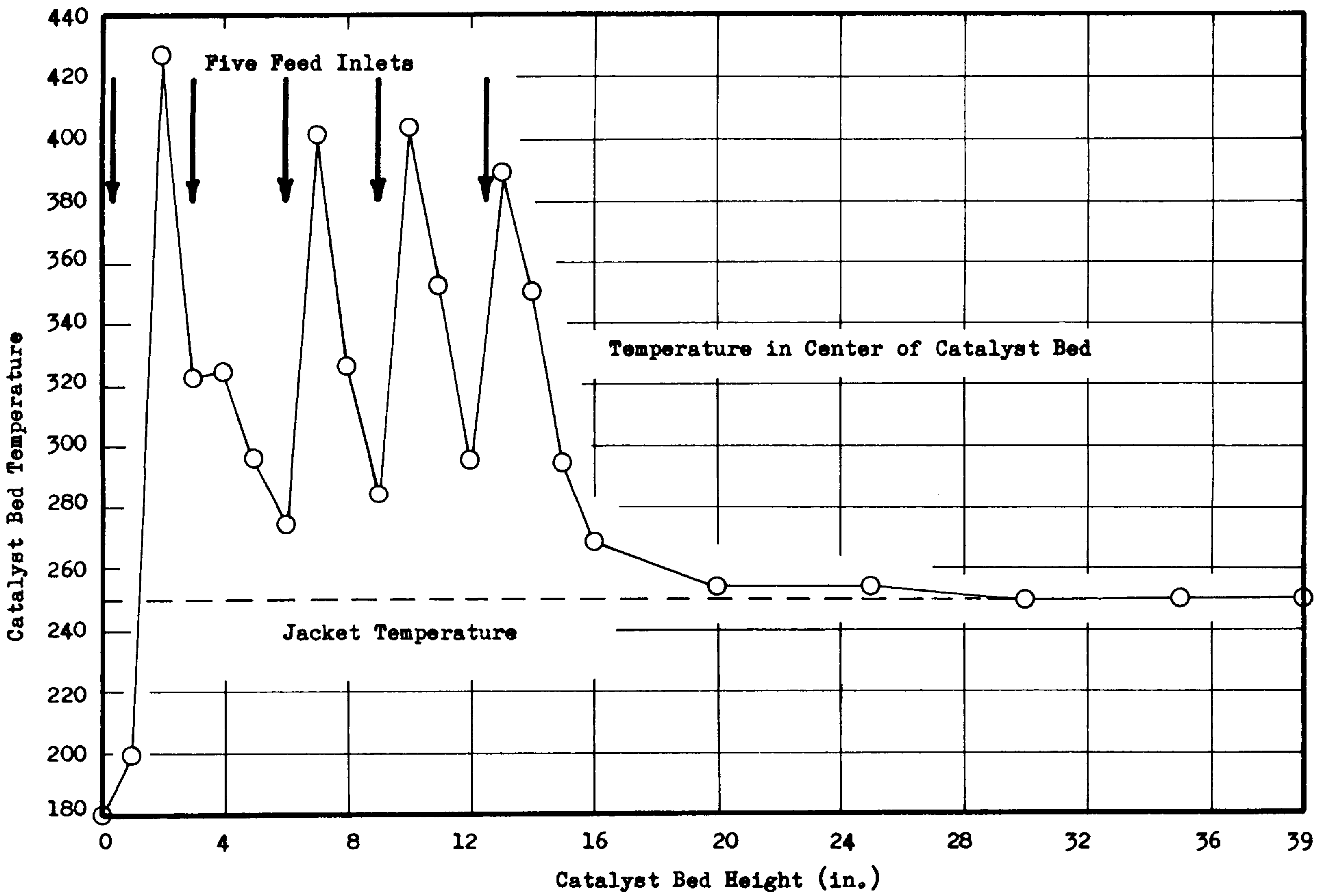


Date = 12-11-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm, Space Velocity = 2500 hr^{-1}, Catalyst 0765-1001-1

Feed Distribution: 0"-20%, 3"-20%, 6"-20%
9"-20%. 12.5"-20%

Catalyst Bed Temperature Profile After 1445 Hours Operation

Figure 17

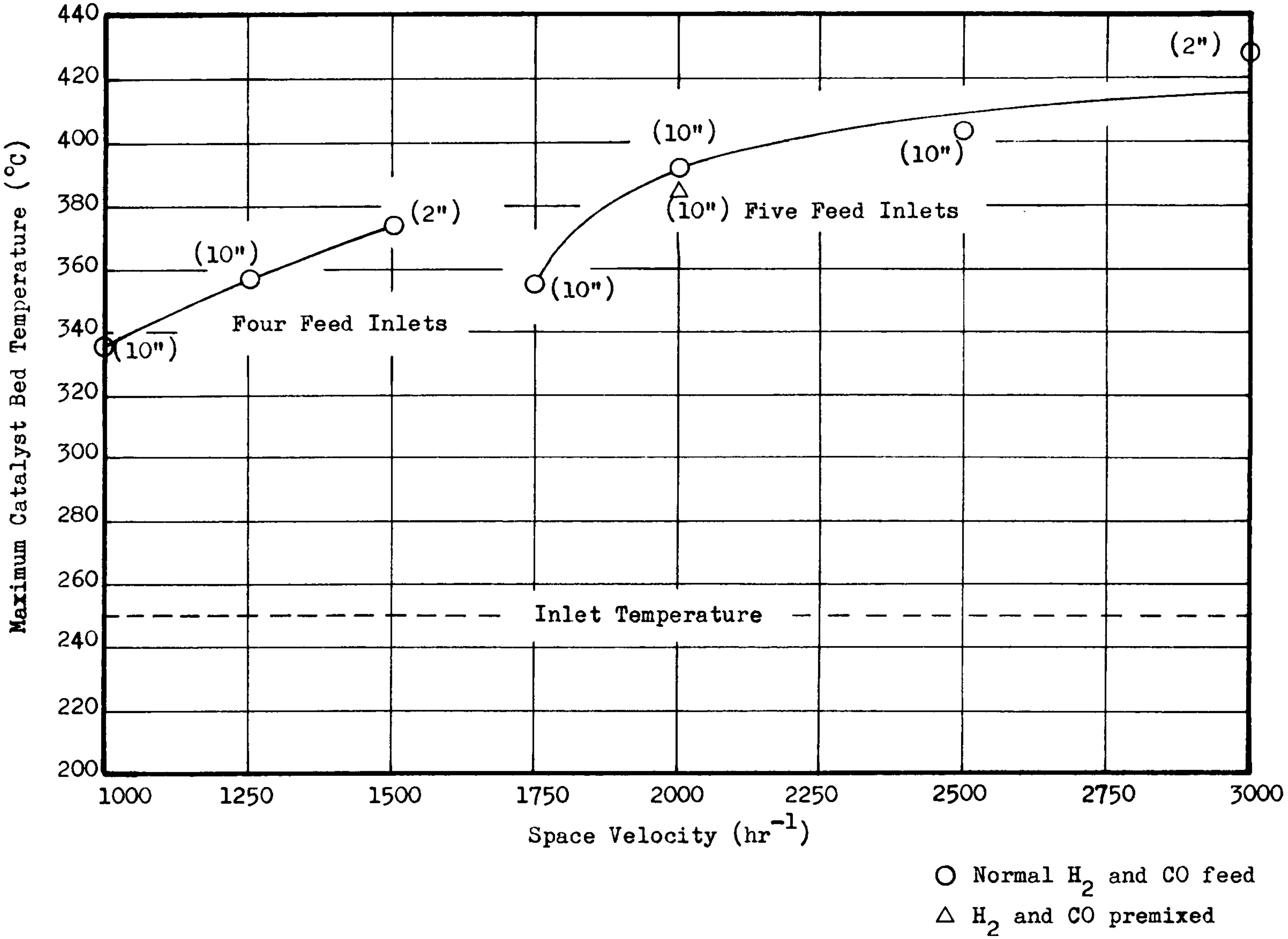


Date = 12-30-4, H_2/CO Mole Ratio = 3.1, Pressure = 6.0 atm., Space Velocity = 3000 hr^{-1}, Catalyst 0765-1001-1.

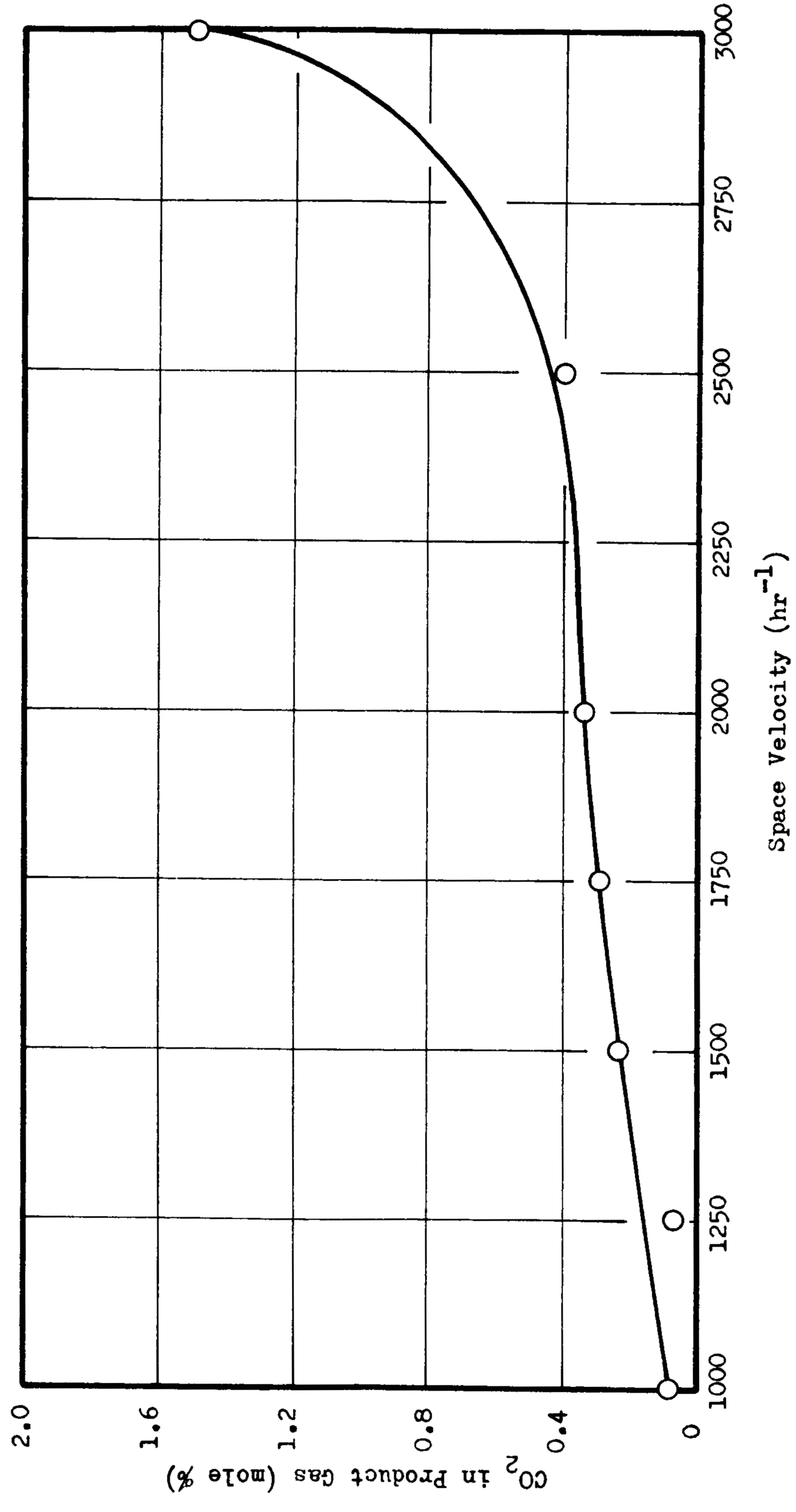
Feed Distribution: 0"-20%, 3"-20%, 6"-20%
9"-20%, 12.5"-20%.

Catalyst Bed Temperature Profile After 1495 Hours Operation

Figure 18

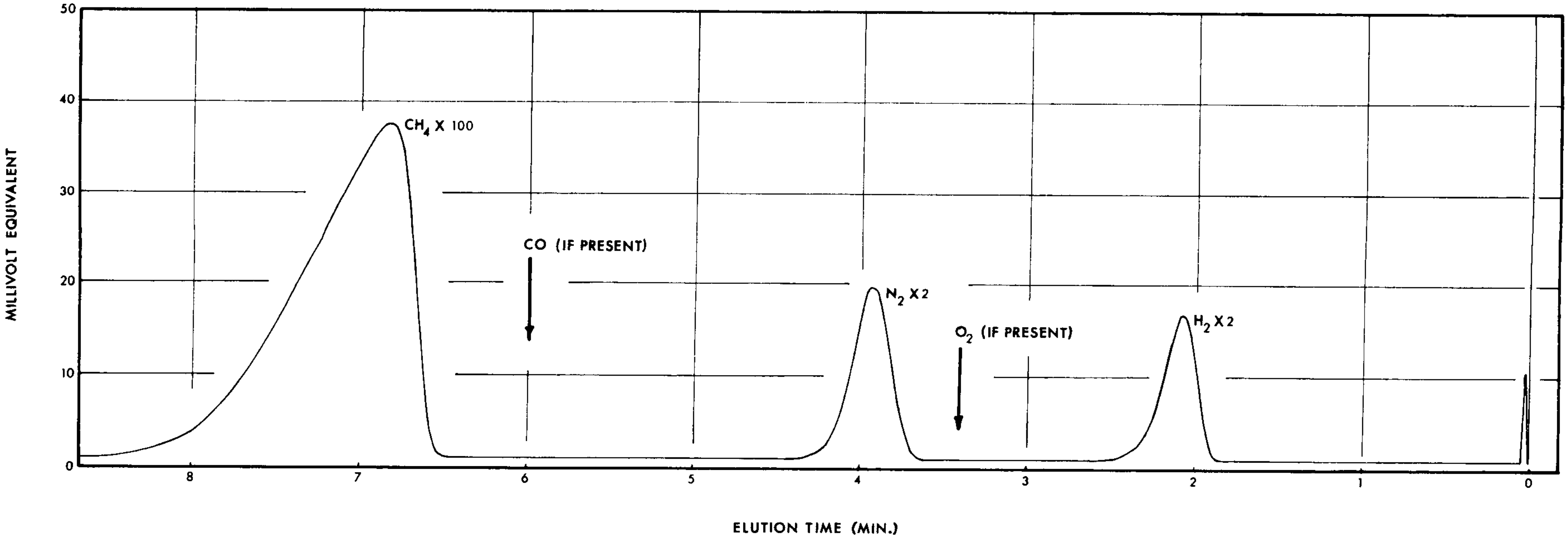


Variation in Catalyst Bed Temperature with Space Velocity
at Constant H_2/CO Mole Ratio



Variation of CO_2 Concentration in Product Gas with Space Velocity at Constant H_2/CO Mole Ratio

Figure 20



Typical Gas Chromatogram of Product Gas at H_2/CO Mole Ratio of 3.1

Figure 21

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