



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.

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Contract NAS 7-225

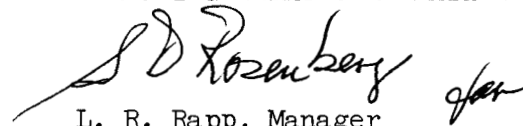
Report No. 0765-03-1 (Quarterly)

October 1964

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 July through 15 October 1964.

AEROJET-GENERAL CORPORATION

A handwritten signature in cursive script, appearing to read "L. R. Rapp", with a small flourish at the end.

L. R. Rapp, Manager
Chemical Products Division

ABSTRACT

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Laboratory apparatus has been placed in operation for studying two of the important steps in the Aerojet Carbothermal Process. A silicate reduction unit was designed, fabricated, and tested. The unit will handle a charge of 1 lb of silicate material, and utilizes a resistance-heated furnace. An automatically controlled carbon monoxide reduction reactor was put into operation for long-term catalyst life studies. This unit gives quantitative conversion of carbon monoxide with excellent water and methane yields. To date, it has operated for more than 600 hours without any apparent change in catalyst activity.

Author

CONTENTS

	<u>Page</u>
I. OBJECTIVE _____	1
II. SUMMARY _____	1
III. TECHNICAL DISCUSSION _____	2
A. Background _____	2
B. Task 1, Carbothermal Reduction of Silicates _____	2
C. Task 2, Carbon Monoxide Reduction Methane Synthesis _____	4
IV. FUTURE WORK _____	8
V. PERSONNEL _____	8
VI. EXPENDITURES AND COMMITMENTS _____	8
References _____	9

	<u>Table</u>
Reduction of Carbon Monoxide with Hydrogen _____	1
Product Gas Analysis _____	2
Carbon Monoxide Conversions and Product Yields _____	3

	<u>Figure</u>
Silicate Reduction Furnace _____	1
Flow Diagram for Silicate Reduction Unit _____	2
Flow Diagram for CO-H ₂ Reduction Unit _____	3
Catalyst Tube for Reduction of CO with H ₂ before Assembly _____	4
Catalyst Tube with Water Jacket _____	5
Unit for CO-H ₂ Reduction for Long Term Operation _____	6
Back Side of CO-H ₂ Reduction Unit _____	7
Catalyst Bed Temperature Profile H ₂ /CO Mole Ratio = 4:1 _____	8
Catalyst Bed Temperature Profile H ₂ /CO Mole Ratio = 3.5:1 _____	9
Distribution List	

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

Research under Extension 2 of Contract NAS 7-225 was initiated on 16 July 1964. The experimental work is to be completed on 15 July 1965. The program is divided into three tasks: Task 1, Carbothermal Reduction of Silicates; Task 2, Carbon Monoxide Reduction Methane Synthesis; and Task 3, Reports.

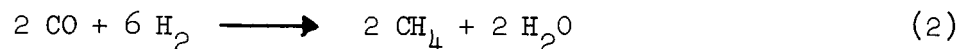
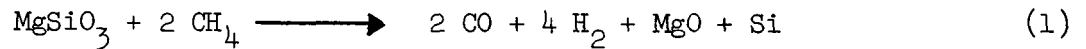
Under Task 1, a silicate reduction unit was designed, fabricated, and tested. The unit will handle a charge of approximately 1 lb of silicate material (granite or acid basalt) utilizing a resistance-heated furnace. The gas inlet tubes were designed using the experience gained during the second program period. The unit has been assembled, leak tested, and brought up to temperature (1650°C).

Under Task 2, the carbon monoxide reduction reactor was modified in a number of ways to achieve automatic operation for long-term catalyst life studies of 60- to 90-day duration. Greater flexibility in the choice of operating conditions was also made possible by the modifications. The reactor was set in operation using a 4:1 H₂/CO mole ratio and a 220°C jacket temperature. Essentially quantitative conversion of carbon monoxide was achieved over a period of 10 days of continuous operation. The conditions were then changed to a 3.5:1 H₂/CO ratio and 250°C jacket temperature. The run was continued for 14 days; excellent conversion and product yields were obtained.

III. TECHNICAL DISCUSSION

A. BACKGROUND

Research under the second extension of Contract NAS 7-225 was started on 16 July 1964. The initial research period was devoted to a study of Step 2 of the Aerojet Carbothermal Process for the manufacture of oxygen from lunar minerals (see Equations 1 through 3 plus Reference 1). The next research period was devoted to a study of Step 1 of the process (Reference 2). The present research is being devoted to an extension of the studies of Steps 1 and 2.



B. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

1. Design and Fabrication of Reactor

The silicate reduction unit was designed to reduce a charge of approximately 1 lb of silicate material (granite or acid basalt) utilizing a resistance-heated furnace. The gas inlet tubes were designed utilizing the information gained during the second program period. This new design is expected to overcome the difficulties encountered previously.

Figure 1 is a cross-sectional assembly drawing of the silicate reduction furnace which was fabricated for the present program. The steel case is 20 in. OD by 26 in. deep, and is designed to withstand a full vacuum. Approximately 6.5 in. of insulation is provided to minimize heat losses and to keep the steel case cool. High-grade zirconia was used to fabricate the gas inlet bells (0.75-in. OD), crucible (2.5-in. OD), heater core (4-in. OD), and outer shield (6-5/8-in. OD). Pure alumina (99%+) was used to fabricate the

gas inlet tubes (3/8-in. OD, 7/32-in. OD, and 1/8-in. OD). Molybdenum was used for the heater element. Vacuum-tight seals were fabricated from rubber stoppers for the optical pyrometer sight tube, electrical heater leads, and gas inlet and outlet tubes. The 9.5-in. ID opening in the furnace top is sealed with an O-ring. Zirconia tubes (not shown) are used to support the crucible and shields. Provisions were made to allow the gas inlet tubes to be raised out of the molten slag without cooling the furnace. This will allow repeated use of the same inlet tubes.

Figure 2 is a flow diagram for the silicate reduction unit. Provisions were made to evacuate and purge the system with argon, to meter methane or methane-hydrogen mixtures into the rock, and to meter and analyze the product gases. Hydrogen-cooling of the inlet tubes is provided. A time-cycle-controller is provided to control the gas flow into and out of the three inlet tubes. This is required to remove the carbon which is slowly deposited on the walls of the inlet bells. A precision optical pyrometer is provided to measure the temperature at the middle of the crucible and at the top of the inlet bells. Several Chromel-Alumel thermocouples (not shown) were installed to measure the temperature at various locations of the inlet tubes. The product gas rate is measured by a wet-test meter. Water and carbon dioxide in the product gas is measured by absorption tubes. Gas analysis is provided by an on-stream gas chromatograph. In addition, these gas analyses will be checked periodically by mass spectrophotometry.

2. Testing of Reactor

Fabrication of the equipment shown in Figures 1 and 2 was completed during this report period. The various lines and systems were tested for leaks with 5 psig argon gas, and under vacuum. All leaks were eliminated. Pressure controllers, flow controllers, and the time-cycle controller were checked out.

The initial heating test of the furnace was made with a small crucible and a single inlet tube. This run demonstrated that the furnace is capable of attaining temperatures of 1650°C or higher. However,

several modifications must be made because the amperage required was higher than anticipated. The molybdenum heater is wound on a zirconia crucible which is conductive above 1200°C. Therefore, higher currents are obtained. For this reason, larger conductive leads to the heating coil will be installed. The 6.5 in. of insulation was found to be insufficient to maintain the steel case below 200°F. Therefore, some water cooling of the case and the connecting lines will be installed. The zirconia shield, zirconia heating crucible, and the melting crucible will be shortened so that additional shielding and insulation can be installed to reduce heat loss from the top of the furnace.

C. TASK 2, CARBON MONOXIDE REDUCTION METHANE SYNTHESIS

1. Modification of Reactor

The carbon monoxide reduction reactor was modified to operate automatically for long-term catalyst life studies of 60- to 90-day duration. The revised unit is much simpler to operate and provides closer temperature control and a greater flexibility in the choice of operating conditions than the one developed during the first program period. As described below, provisions were made to measure temperatures at any catalyst bed level. Multiple entry for reactant gas is another notable modification which provides uniform bed temperatures throughout the reaction zone. This should result in extended catalyst life and permit the use of higher space velocities.

Figure 3 is a flow diagram of the carbon monoxide-hydrogen reduction unit. Hydrogen and carbon monoxide are obtained from commercial cylinders. A constant pressure (200 psig) is provided on the orifice meters by standard gas cylinder pressure reducing regulators. Line filters are provided to remove dirt from the gases. All lines and fittings are of stainless steel to prevent corrosion and contamination. Solenoid valves are installed in both feed lines to automatically shut down the unit in case of high catalyst bed temperature, low gas pressure (line break), or failure of electrical power or water. Gas rates are measured and controlled by calibrated flow recording controllers.

Uniform and controlled catalyst bed temperatures are provided by a water jacket (2-in. ID) around the catalyst bed. The exothermic reaction between the gases entering the bottom of the reactor generates steam bubbles in the water which rise and maintain a uniform jacket temperature. The jacket temperature is very closely controlled by means of a pressure controller which maintains a constant temperature in the jacket by regulating the flow of water in the cooling coil.

Heat for catalyst regeneration, startup, and/or isothermal operation is provided by two 3/16-in. OD heater tubes (not shown) extending up throughout the length of the water jacket. The carbon monoxide or mixed carbon monoxide-hydrogen stream is distributed as desired at the 0, 3, 6, or 9-in. level (from the bottom of the catalyst bed) by means of rotameters and needle valves. The outlet gases are cooled and condensed in a coil of 0.25 in. stainless-steel tubing immersed in cooling water. Safety pressure release is provided by a relief valve on the water jacket and by a mercury release bubbler on the low-pressure product gas line.

The product water is separated from the gas in a 250-ml stainless-steel sample container. The level in the water separator is maintained constant by a solenoid valve (not shown) which is actuated by a relay connected to a mercury contact in a manometer. The pressure in the catalyst bed can be controlled accurately at 1 to 10 atmospheres by means of a pressure controller and motor valve installed in the product gas line. The product gas from the 12.5-, 19-, 25.5-, 32-, and 39-in. catalyst bed level is analyzed by an on-stream gas chromatograph, an Orsat, or a mass spectrophotometer. A manometer (50 in. of mercury) is used to measure the pressure drop across the catalyst bed. The product gas flow rates are metered by a wet-test meter.

Figure 4 is a photograph of the catalyst tube before assembly. The unit is fabricated from a 5/8-in. OD stainless-steel tube with 1.25-in. OD stainless-steel fins. The inlet tubes, sample lines, drain line, vent line, jacket thermocouple tube, cooling coil, and lower and upper flanges are welded to the catalyst tube.

Figure 5 is a photograph of the catalyst tube with the water jacket installed. In addition to the connections shown in Figure 5, the 2-in. stainless-steel pipe water jacket, and the 3/16-in. OD heater tubes are shown in this photograph. The AN fittings on the ends of the catalyst tube are shown as well.

Figure 6 is a photograph of the completed carbon monoxide-hydrogen reduction unit. On the panel board can be seen the pressure and flow controllers, multipoint temperature recorder, Variacs for jacket and line heater control, and pressure gages. The reactor (complete with a 3-in. layer of insulation) and the insulated condenser and water separator are also visible. The muffle-type furnace was installed for hydrogen reduction of the catalyst; this furnace is removed during carbon monoxide reduction runs.

Figure 7 is a photograph of the back side of the unit showing the gas cylinder, pressure-reducing regulators, manometers, differential pressure transfer cells, and the gas chromatograph.

2. Long-Duration Runs

The equipment was leak-tested and the first shakedown test run was started. This run was made under the following conditions: (a) catalyst type 0765-1001 (nickel on kieselguhr) crushed to -9 to +16 mesh; (b) 1000 space velocity (based on 119 cu cm of catalyst); (c) 4:1 H₂/CO mole ratio; (d) 6 atm pressure in catalyst bed; (e) 220°C jacket temperature; and (f) feed entry split equally at 0, 3, 6, and 9 in. Under these conditions, 100% conversion of the carbon monoxide was obtained (no CO or CO₂ in outlet gas) with about 0.1 in. mercury pressure drop across the catalyst bed and a catalyst-bed-temperature profile as shown in Figure 8. There was a rapid increase in temperature at each point of feed entry, after which the gases tended to cool to the temperature of the water jacket.

The conditions for the first two runs are given in Table 1 showing the time of operation, flow rates, pressures, and temperatures. The product gas analyses are listed in Table 2. No carbon monoxide or carbon dioxide were ever detected during either run.

Table 3 presents additional data on reaction conditions and product yields. The 100.0% values of carbon monoxide conversion are based on the absence of carbon monoxide in the product gas. The nominal product yields are based on the assumption of a 100% oxygen material balance. The lower actual yields of water and methane are ascribed to loss of reactant and/or product gas from the system caused by very small leaks.

Run 1 was continued for 238 hours with only short duration interruptions for equipment maintenance. There was no detectable loss of catalyst activity during this period as indicated by product analysis and unchanging temperature profile and pressure drop across the catalyst bed. These results confirmed the findings and predictions made with the same catalyst operating with similar conditions in the first program (Reference 1).

Run 2 was started with slightly more stringent conditions (3.5:1 H₂/CO ratio). The catalyst bed pressure and space velocity were maintained constant as in the first run. The average catalyst bed temperature was raised slightly (to 250°C) in order to more closely approximate the earlier work. This run was continued for 333 hours. Once again, the outlet gas contained less than 0.001% of carbon monoxide or carbon dioxide. The catalyst bed temperature profiles (see Figure 9) were quite similar to the one for Run 1, and showed no signs of catalyst deactivation.

Because of the importance of obtaining quantitative conversion in the carbothermal process, the material balances and yields are based on measurements and analyses using accurately calibrated equipment. Methane yields are based on gas chromatographic (gc) analyses, calibrated wet-test meter flows, and the weight of carbon monoxide consumed over a given time period. Water yields are based on weight of water produced and weight of carbon monoxide consumed over a given time period. The water analyses deviate $\pm 2.6\%$ from a mean value, and the methane yields deviate $\pm 4.2\%$ from the mean. These deviations reflect the momentary variation in flow rates as well as inherent errors in the analytical procedures. Variations in flow rates tend to average out over a long period of time. The errors inherent in the water analyses are estimated to be less than $\pm 1\%$, and the gc errors are less than $\pm 2\%$. The calibration of the gc apparatus is frequently checked with a standard sample of methane.

IV. FUTURE WORK

A. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

Tests with the furnace containing a charge of rock will be made to determine heating time and obtainable temperatures. Experiments will then be conducted on the reduction of silicate rock (granite and rhyolite) and tektites with methane gas. Initial experiments will be directed toward quantitative utilization of methane carbon.

B. TASK 2, CARBON MONOXIDE REDUCTION METHANE SYNTHESIS

Long-duration runs will be continued using a 3.1 to 1 H₂/CO mole ratio. Material balances and bed temperature profiles will be frequently checked to determine any change in catalyst activity.

C. TASK 3, REPORTS

Reports will be submitted as scheduled.

V. PERSONNEL

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

VI. EXPENDITURES AND COMMITMENTS

Approximately 1475 man-hours and \$21,000 were expended on Contract NAS 7-225 during this report period.

REFERENCES

1. S. D. Rosenberg, G. A. Guter, F. E. Miller, and G. R. Jameson, Research on Processes for Utilization of Lunar Resources, Aerojet-General Summary Report No. 2757, Contract NAS 7-225 (December 1963); see also S. D. Rosenberg, G. A. Guter, F. E. Miller, and G. R. Jameson, Catalytic Reduction of Carbon Monoxide with Hydrogen, NASA Contractor Report NASA CR-57 (July 1964).
2. 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).

REDUCTION OF CARBON MONOXIDE WITH HYDROGEN
(OPERATING DATA)^a

Run No.	Period of Operation (hours)	Reactant Flow Rate		Product Flow Rate		Pressure		Temperature		
		H ₂ (g mole/hr)	CO (g mole/hr)	Gas (g mole/hr)	Liquid Water (g/hr)	Water Jacket (psig)	Static (psig)	Catalyst Bed ΔP (In. Hg)	Water Jacket (°C)	Catalyst Bed Maximum ^b (°C)
1 a	26-75	3.93	0.986	1.8649	-	350	76.0	0.1	220	275
1 b	75-176	3.93	0.986	1.8454	16.12	360	75.5	0.1	223	296
1 c	176-238	3.93	0.986	1.6208	16.6	355	76.0	0.1	222	299
2 a	0-17	3.39	1.09	1.5661	19.65	565	75.0	0.1	249	331
2 b	17-41	3.39	1.09	1.6677	18.24	565	75.5	0.12	248	328
2 c	41-65	3.39	1.09	1.5964	18.24	560	76.0	0.12	248	326
2 d	65-82	3.39	1.09	1.6179	18.25	575	76.0	0.11	250	327
2 e	82-103	3.39	1.09	1.6130	18.23	575	76.5	0.15	249	330
2 f	103-148	3.39	1.09	1.6042	19.08	600	77.0	0.18	253	335
2 g	148-169	3.39	1.09	1.5925	19.05	580	76.5	0.17	251	332
2 h	169-194	3.39	1.09	1.5935	19.21	570	74.5	0.13	250	331
2 i	194-218	3.39	1.09	1.6023	18.95	570	75.5	0.13	249	329
2 j	218-237	3.39	1.09	1.5232	19.35	580	75.5	0.16	250	331
2 k	237-261	3.39	1.09	1.4060	19.49	570	76.0	0.15	250	333
2 l	261-333	3.39	1.09	1.5661	19.17	580	76.0	0.16	247	330

^a Using Catalyst 0765-1001-1 with the CO feed equally divided among the 0, 3, 6, and 9 in. feed lines.

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

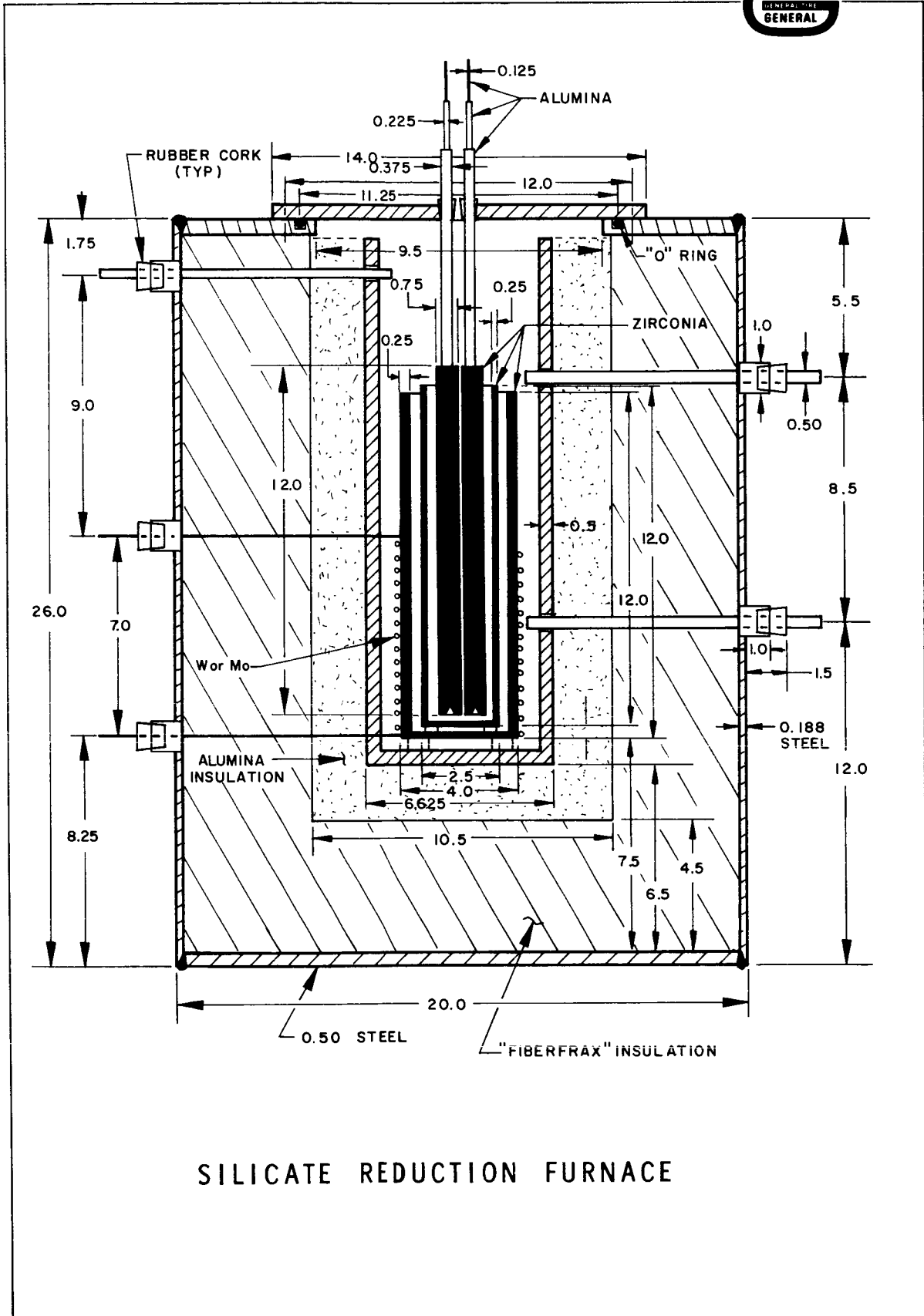
PRODUCT GAS ANALYSIS

Run No.	Composition of Product Gas (Vol %)				
	H ₂	CO	CH ₄	CO ₂	H ₂ O
1	(49.7) ^a	0.00	(49.8) ^a	0.00	0.48
2 a	26.2	0.00	73.2	0.00	0.48
2 b	35.6	0.00	63.7	0.00	0.46
2 c	35.7	0.00	61.1	0.00	0.44
2 d	36.5	0.00	62.6	0.00	0.44
2 e	30.2	0.00	68.8	0.00	0.44
2 f	34.7	0.00	64.6	0.00	0.44
2 g	34.1	0.00	65.2	0.00	0.44
2 h	31.4	0.00	67.8	0.00	0.44
2 i	30.2	0.00	69.0	0.00	0.44
2 j	31.9	0.00	67.5	0.00	0.44
2 k	31.6	0.00	67.7	0.00	0.44
2 l	30.3	0.00	69.1	0.00	0.44

^a Average value for the complete run

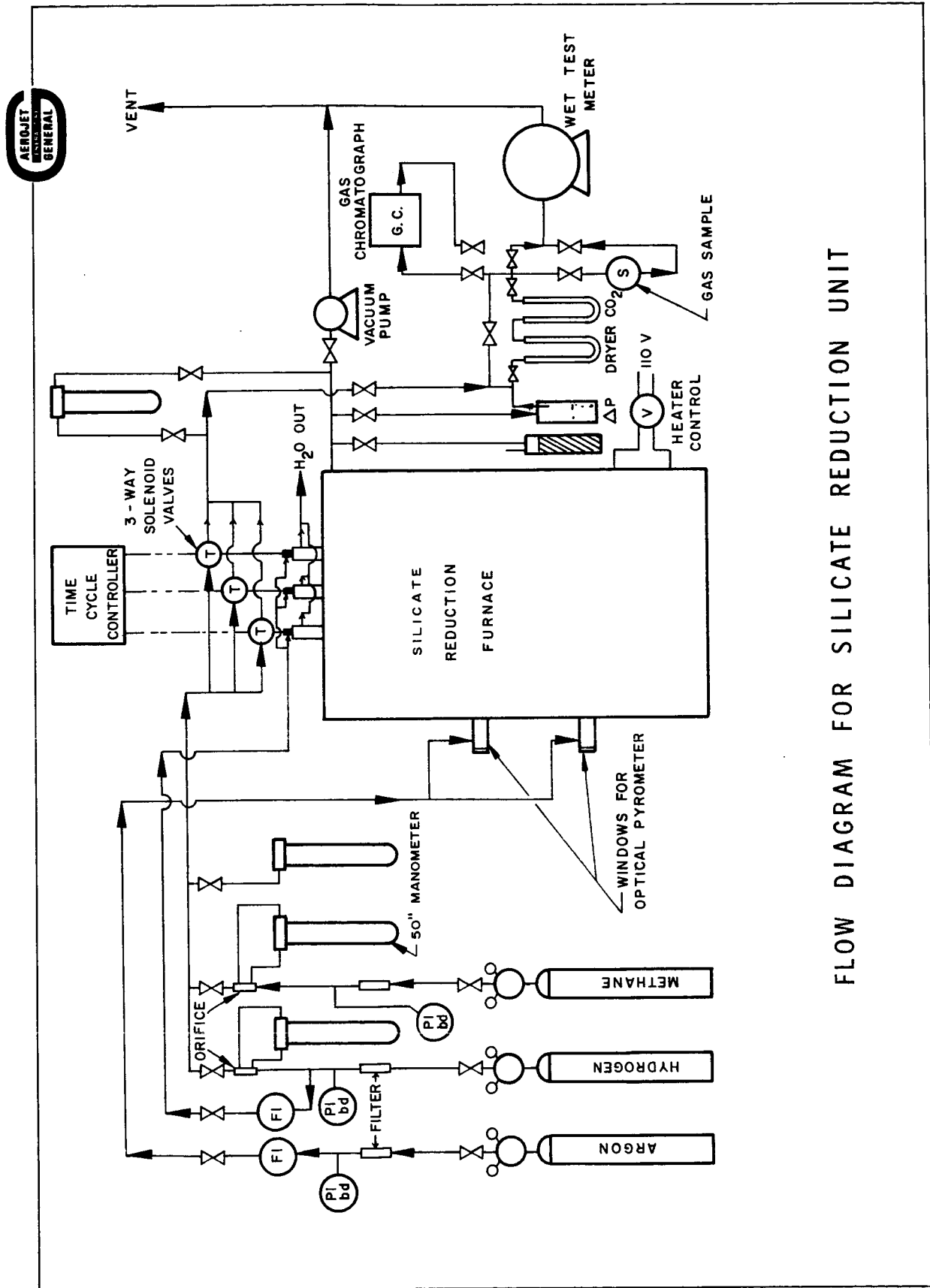
CARBON MONOXIDE CONVERSIONS AND PRODUCT YIELDS

Run No.	Nominal H ₂ /CO Mole Ratio	Space Velocity (hr ⁻¹)	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		Normalized Product Yield (mole %)	
					H ₂ O	CH ₄	H ₂ O	CH ₄
1 a	4:1	1000	220	100.0	-	-	-	-
1 b	4:1	1000	220	100.0	92.48	98.6	92.48	98.6
1 c	4:1	1000	220	100.0	94.84	100.5	94.84	100.5
				average	93.38	99.55	93.38	99.55
2 a	3.5:1	1000	250	100.0	101.498	105.57	101.498	105.57
2 b	3.5:1	1000	250	100.0	94.431	97.83	94.431	97.83
2 c	3.5:1	1000	250	100.0	94.329	89.82	94.329	89.82
2 d	3.5:1	1000	250	100.0	94.329	93.27	94.329	93.27
2 e	3.5:1	1000	250	100.0	94.329	102.20	94.329	102.20
2 f	3.5:1	1000	250	100.0	98.53	95.43	98.53	95.43
2 g	3.5:1	1000	250	100.0	98.53	95.62	98.53	95.62
2 h	3.5:1	1000	250	100.0	94.23	99.49	94.23	99.49
2 i	3.5:1	1000	250	100.0	97.61	101.81	97.61	101.81
2 j	3.5:1	1000	250	100.0	100.06	94.68	100.06	94.68
2 k	3.5:1	1000	250	100.0	100.78	87.66	100.78	87.66
2 l	3.5:1	1000	250	100.0	99.04	99.66	99.04	99.66
				average	97.602	97.124	97.602	97.124
							100.00	99.51



SILICATE REDUCTION FURNACE

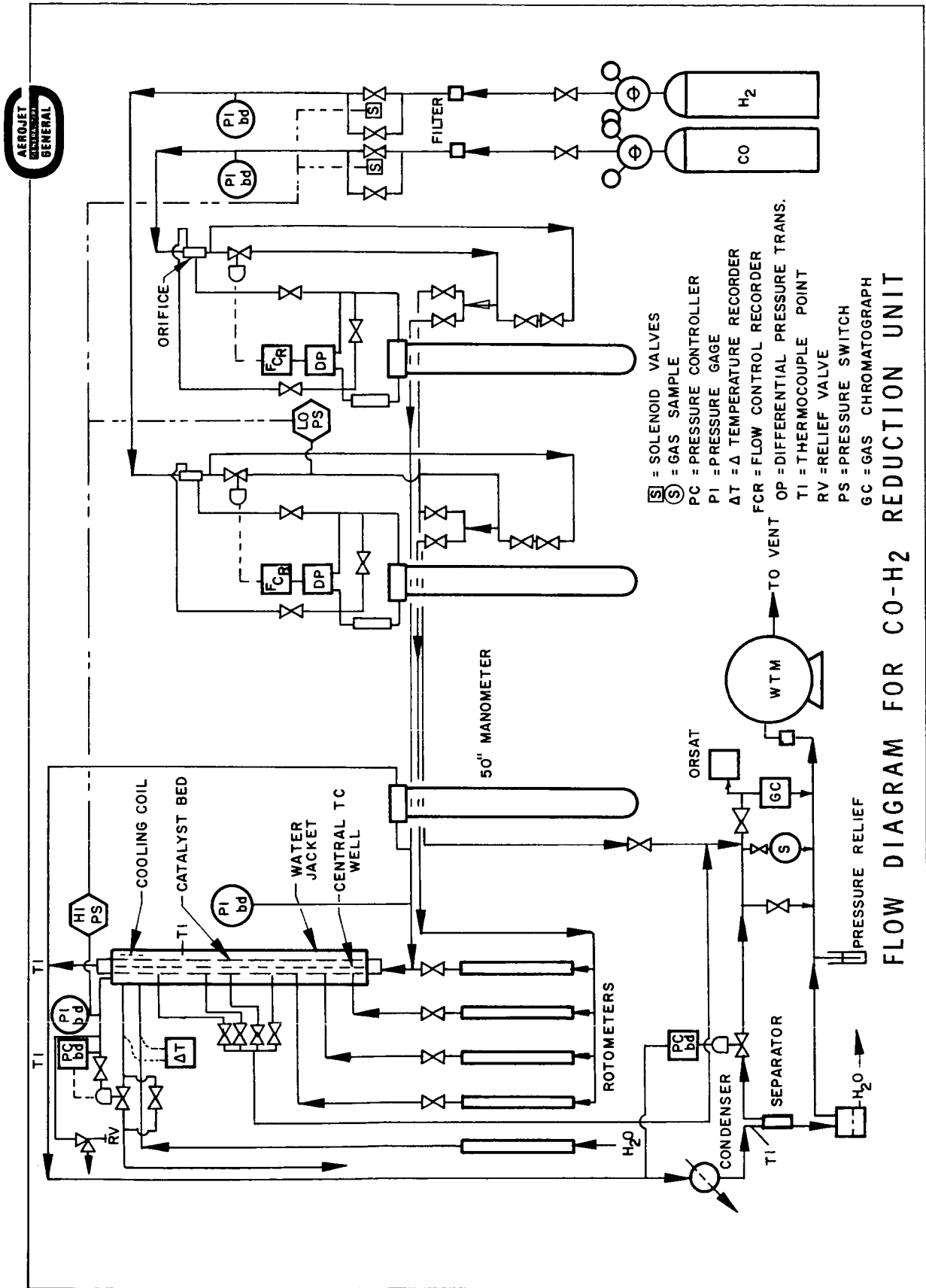
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FLOW DIAGRAM FOR SILICATE REDUCTION UNIT

Figure 2

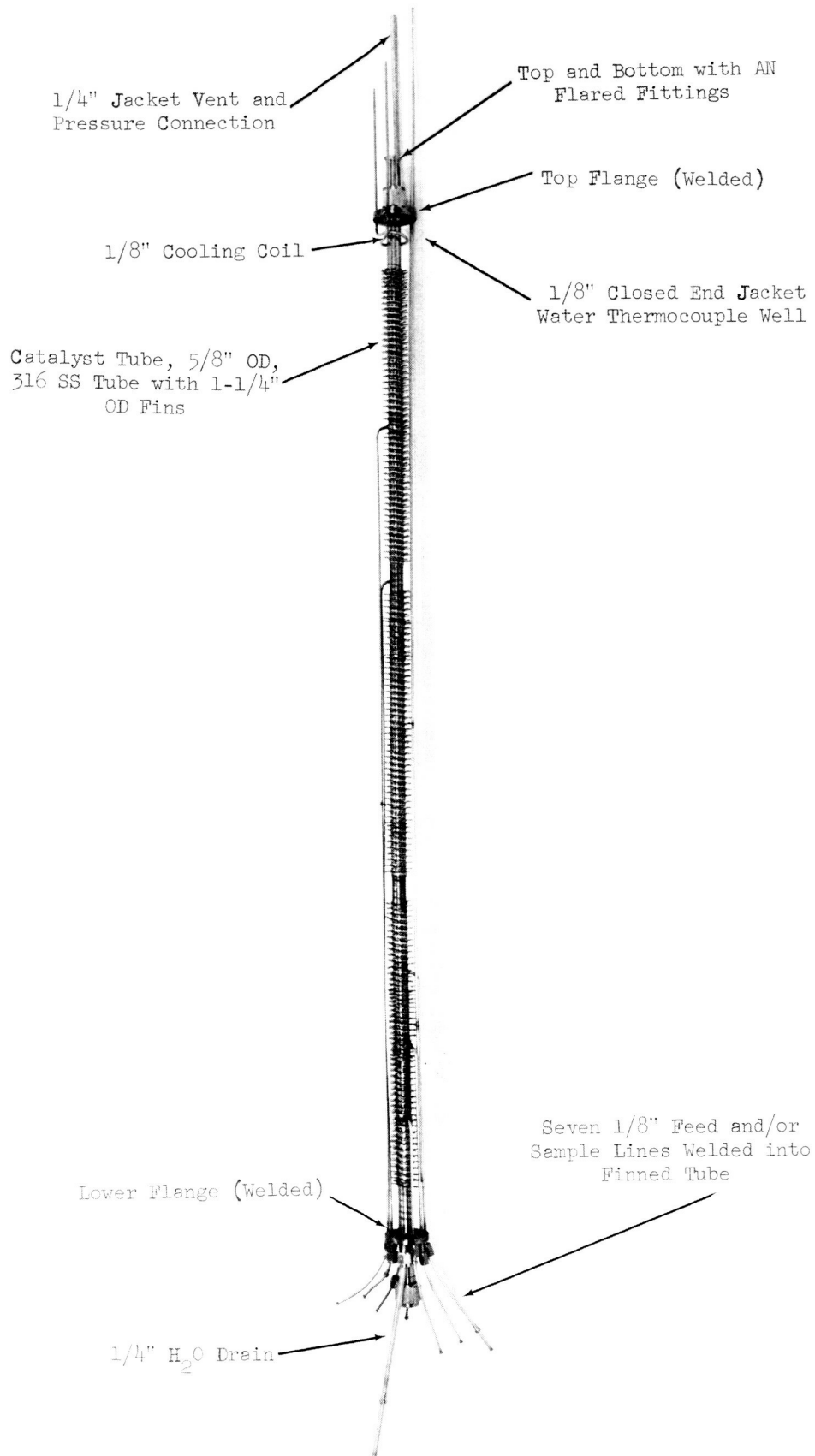
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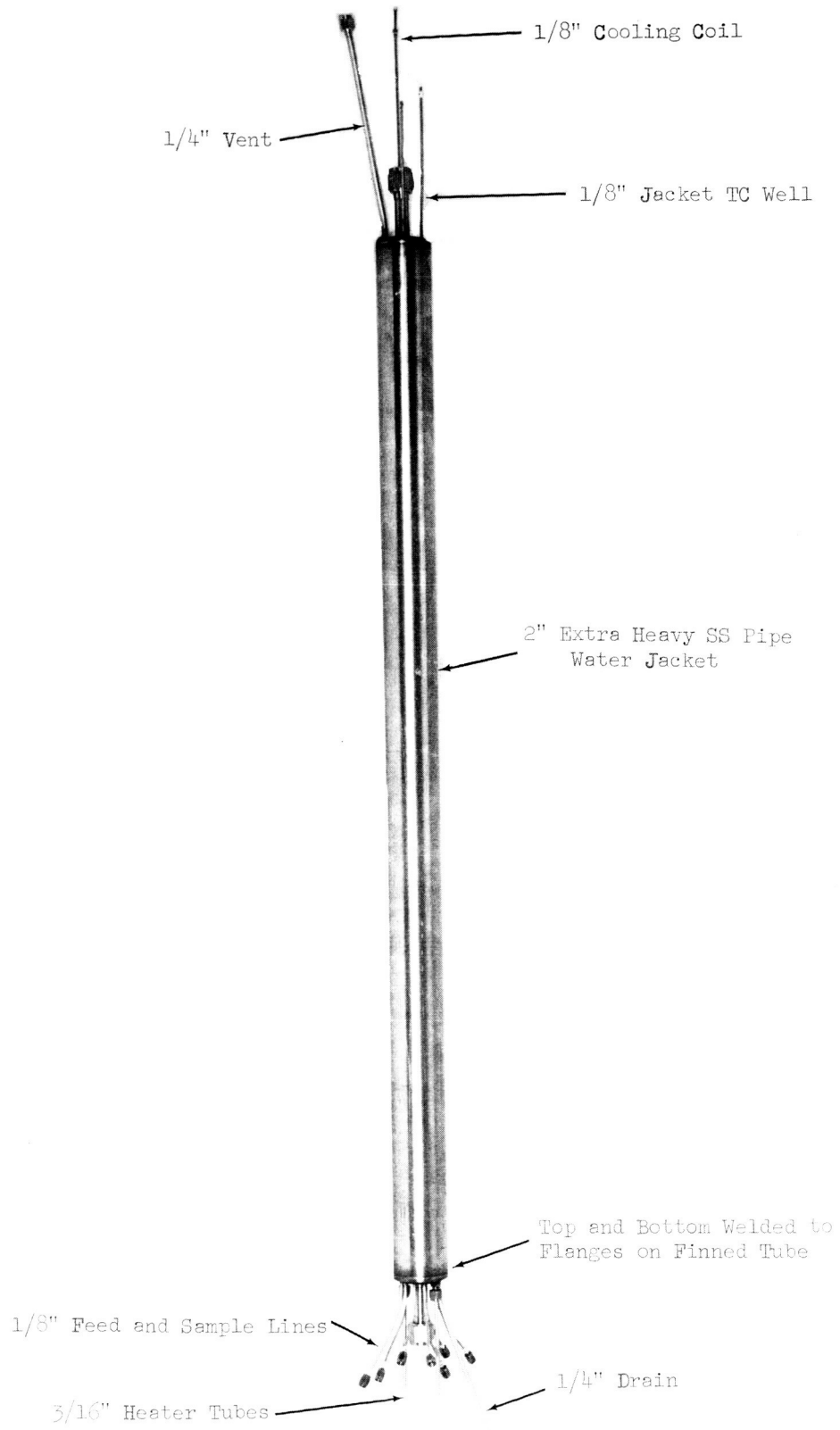
FLOW DIAGRAM FOR CO-H₂ REDUCTION UNIT

10-087-116

Figure 3

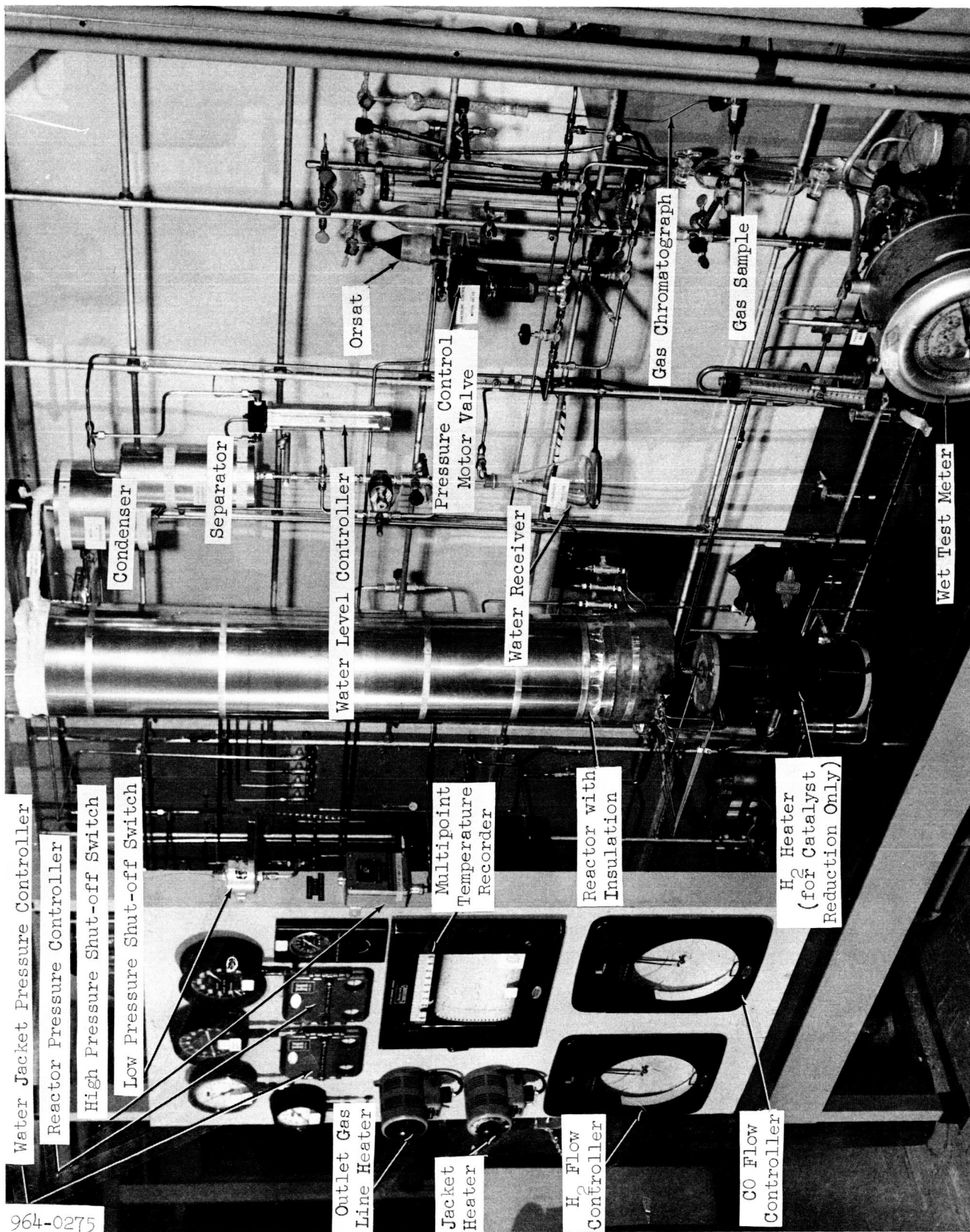


Catalyst Tube for Reduction of CO with H₂ Before Assembly

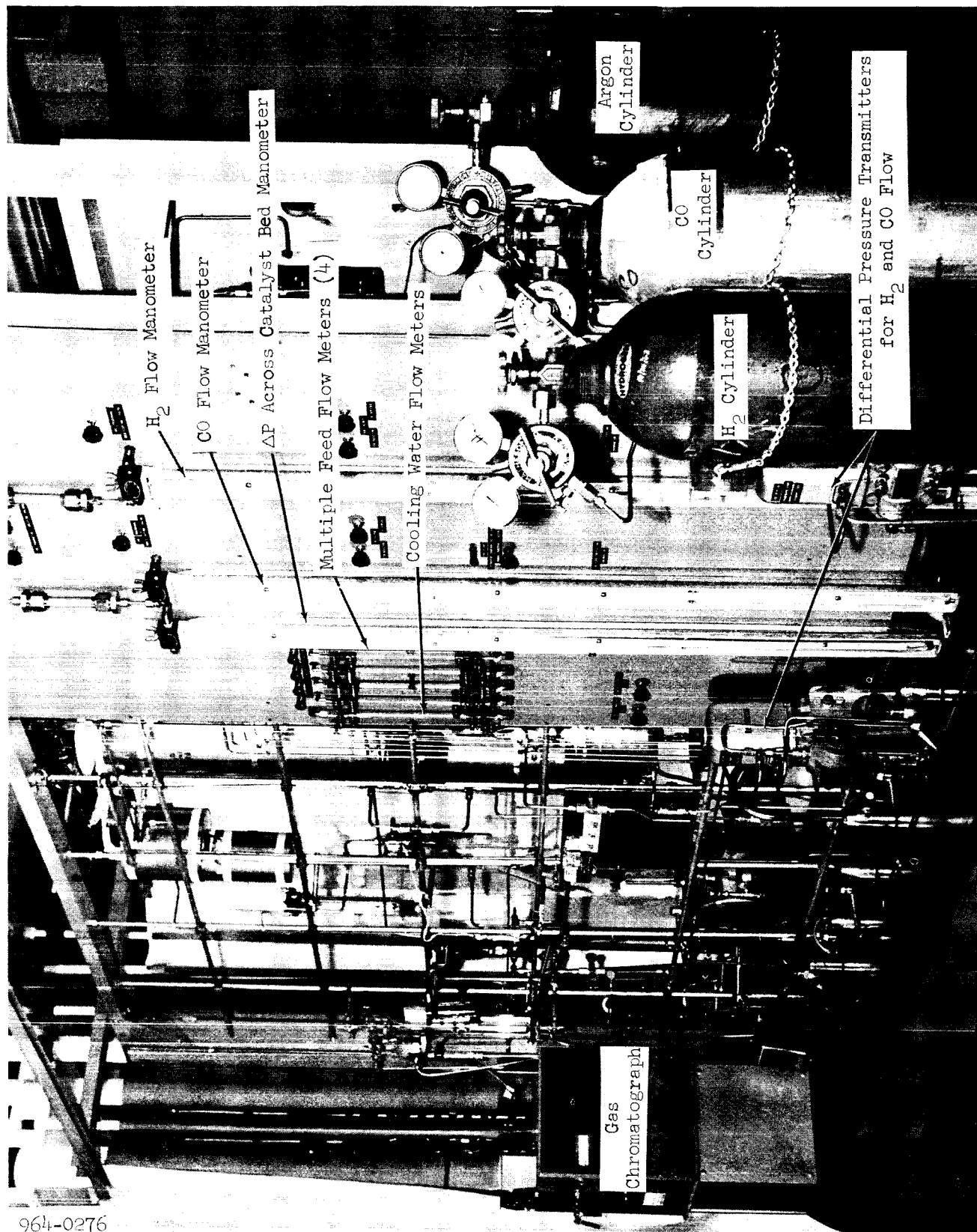


864-0261

Catalyst Tube with Water Jacket



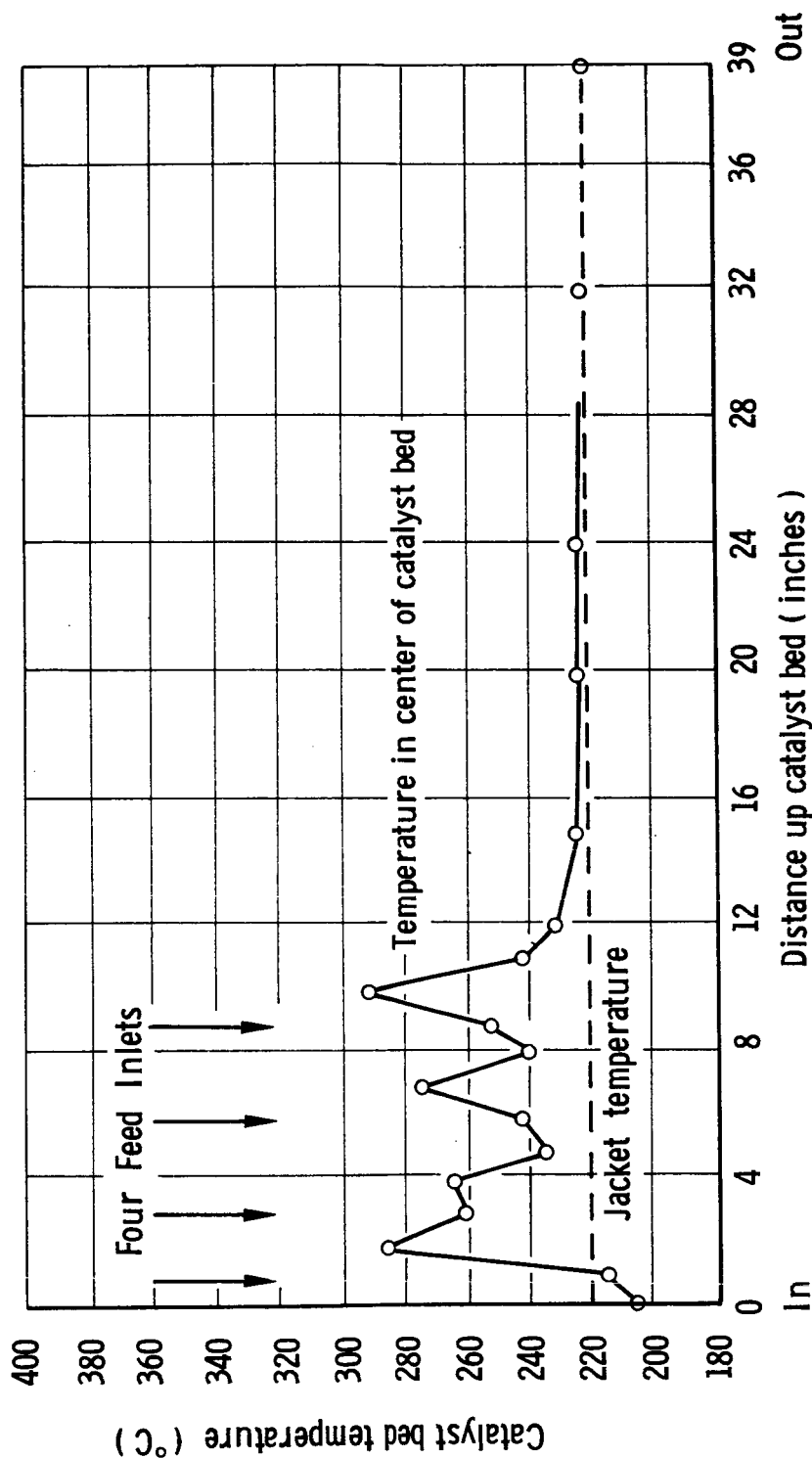
Unit for CO-H₂ Reduction for Long Term Operation



964-0276

Back Side of CO-H₂ Reduction Unit

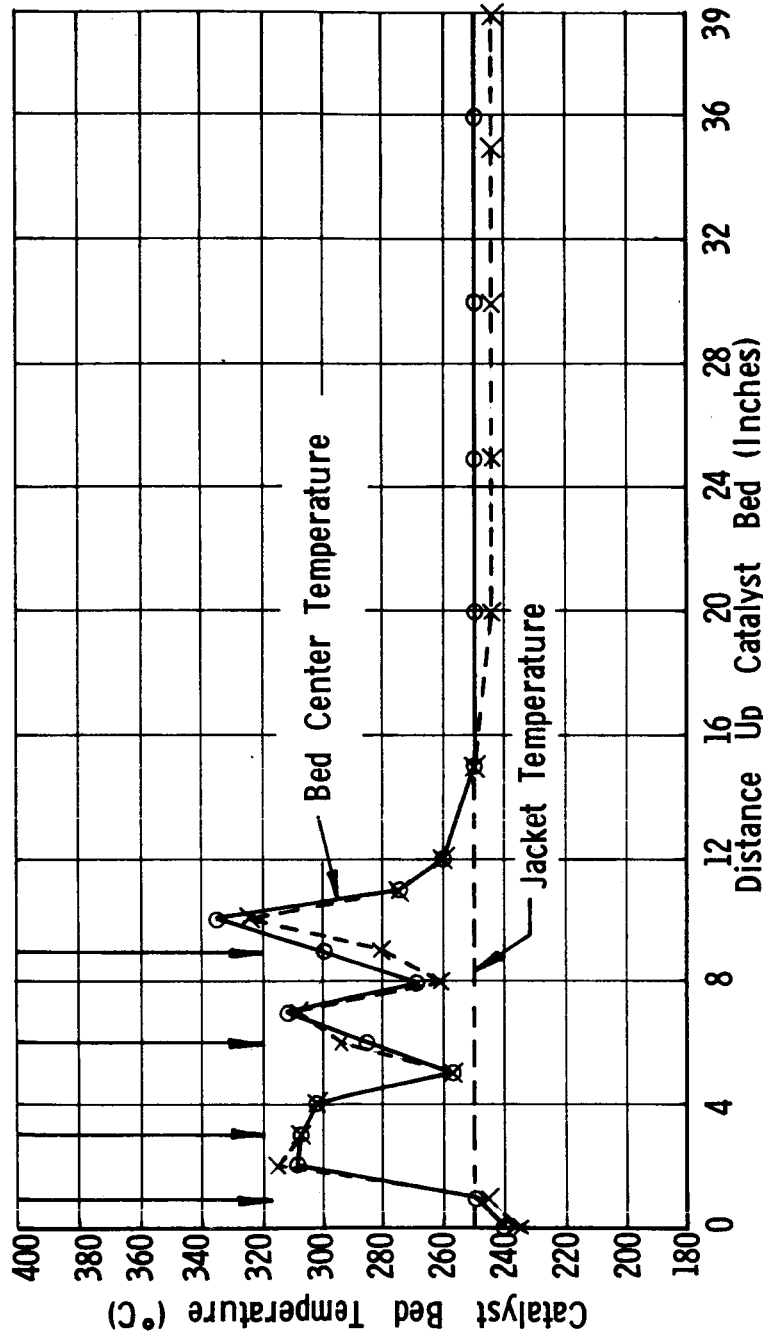
Figure 7



H₂ / CO = 4, Pressure = 6.0 Atm., Space Velocity = 1000, Catalyst 0765-1001-1, Feed Distribution : 0"-25%, 3"-25%, 6"-25%, 9"-25%, After 30 hr operation

CATALYST BED TEMPERATURE PROFILE

10-067-116



H₂/CO=3.5 Pressure 6.0 Atm Space Velocity 1000 Catalyst 0765-1001-1
 Feed Distribution: 0"-25%, 3"-25% ○—○ 5 Oct. After 2hrs at 3.5:1
 6"-25%, 9"-25% ×—× 20 Oct. After 336 hrs at 3.5:1

CATALYST BED TEMPERATURE PROFILE

10-0027-110

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