VON KARMAN CENTER

CHEMICAL PRODUCTS DIVISION

RESEARCH ON PROCESSES FOR UTILIZATION OF LUNAR RESOURCES

A REPORT TO

NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

CONTRACT NAS 7-225

REPORT NO. 0765-03-3 (QUARTERLY) / APRIL 1965 / COPY NO. 31

AEROJET
GENERAL TIRE
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AEROJET-GENERAL CORPORATION

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. 0765-03-3 (Quarterly) April 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 January through 15 April 1965.

AEROJET-GENERAL CORPORATION

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ABSTRACT

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Seven runs were completed with the silicate reduction furnace. Reductions with graphite gave up to 86% conversion of carbon to carbon monoxide, while reducing 50% of the silicate. Reductions with methane indicated that further modification of the methane inlet tubes is required. The second long-duration run (90 days) with the carbon monoxide reduction apparatus was initiated; it has completed 54 days of successful operation, giving excellent conversions and product yields.

Author

CONTENTS

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, seven silicate reduction runs were made during this report period - four using methane and three with graphite as the reducing agents. The runs with methane clearly indicated the need for mechanical removal of the carbon which deposits in the reactant gas inlet tube. Up to 86% of the charged graphite was converted into carbon monoxide. It was also demonstrated that too rapid an evolution of carbon monoxide considerably reduced the efficiency of the reaction.

Under Task 2, the second run of long duration (90 days) was started with the carbon monoxide reduction reactor. After 54 days of operation, the conversion yields and material balances were excellent.

III. TECHNICAL DISCUSSION

A. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

1. Reactor Modifications[*]

Several modifications of the rock reduction furnace were made during this report period.

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[*] See Aerojet-General Quarterly Reports 0765-03-1 and -2, Contract NAS 7-225 (October 1964 and January 1965, respectively) for a complete description of the silicate reduction unit.

a. The cooling coil inside the furnace was moved up from 7 to 4 in. below the top of the furnace, and its size was increased from 4 to 6 turns of 1/4-in. stainless-steel tube. This was done after the coil burned out during Run 6.

b. A 1/8-in.-OD alumina rod was installed in the product gas outlet tube for Runs 8, 9, and 10. This rod entered through a packing gland in one leg of a pipe tee so that the rod could be periodically raised and lowered (1 to 2 in.) in order to prevent the outlet tube from becoming clogged with sublimate.

c. Zirconia tubes (1/2-in.-OD by 18-in.-long) were used for inlet tubes for Runs 8 and 9 instead of the 3/4-in.-OD bells sealed to 1/8-in. tubes used in the earlier runs. This change eliminated the troublesome seal problem.

d. A 0.003-in.-thick molybdenum liner was installed in the reactant gas inlet tube for Run 9. This prevented carbon from reacting with the zirconia tube and thereby eliminated the embrittlement previously experienced.

e. The cycle timer operation for feed gas was modified from a "20 min on, 10 min off" cycle to an "8 min on, 2 min off" cycle and finally to an "8 min on, 50 sec off, 10 sec surge" cycle. This was done in an effort to find a means of removing carbon from the reactant gas inlet tube.

2. Reduction Experiments

Seven silicate reduction runs were carried out during this quarter. Methane was used as the reducing agent in four of the runs; graphite was used in three. Acidic basalt was used as the natural silicate in six of the runs; granite was used in one. The results of these experiments are summarized in Table 1.

a. Runs 6, 7, and 10 (Reduction of Acidic Basalt with Graphite)

Run 6 was made with 1 lb of basalt (-6 to +28 mesh) mixed with 60.5 g of pure graphite (emission spectrograph electrodes) ground to -6 to +28 mesh size. The purpose of this run was to determine if essentially 100% of the carbon could be recovered (as CO) when enough graphite was added to the rock to react with 50% of the silicate.

The data for Run 6 are presented in Table 2 and Figure 1. Although the data points were taken every 30 min, the data are reported in the table at 4-hour intervals due to the length of the run (68 hours). The crucible was heated at the rate of about 60°C/hour, up to about 1400°C (see Figure 1). Carbon monoxide started to evolve at about 1040°C. The basalt melted at about 1200°C. At 1400°C, carbon monoxide started to evolve rather rapidly; the rate of heating was then decreased. Trouble was experienced after 31 hours of operation, at which time the product gas outlet tube (1/2-in. ID) became clogged with sublimate. The furnace heater was turned off for several hours while an alternate gas outlet tube was installed; heating was then resumed. The crucible temperature was gradually increased as the rate of carbon monoxide evolution declined. The run was terminated after 68 hours, at which time 86% of the graphite charged had been recovered as carbon monoxide and a crucible wall temperature of 1785°C had been attained. At shutdown, carbon monoxide was being evolved very slowly (i.e., at a rate of 0.25%/hour).

Inspection of the crucible after the run (see Figures 2 and 3) showed that while it had been severely blistered and somewhat deformed, it had not cracked. Metal and a small amount of slag remained in the crucible. A very small amount of sublimate was found deposited in the outlet tube. The slag was absorbed by the crucible and/or volatilized out of the crucible and was deposited in the insulation of the furnace.

Run 7 was made with 1 lb of basalt (-6 to +28 mesh) mixed with 60.5 g of finely ground graphite (-28 to +48 mesh). This run was a duplicate of Run 6 except that the graphite used was more finely ground. The purpose of this experiment was to determine whether the more finely ground carbon would react faster than the Run 6 carbon and provide a higher carbon monoxide yield.

The results of this run are shown in Table 3 and Figure 4. The reaction did indeed go faster with the more finely ground carbon; 36% of the carbon was recovered as carbon monoxide in 12 hours in Run 7, as compared with 22 hours for the same carbon recovery in Run 6. It soon became apparent that there is an inherent limitation on the rate of gas production using the current apparatus. If this rate is exceeded, foaming or frothing of the melt will result. Shortly after 12 hours of operation, the gas outlet tube clogged. Another gas outlet from the furnace was opened, but thereafter the rate of carbon monoxide production dropped off rapidly.

Examination of the crucible after the run showed that the first outlet tube was clogged with froth from the melt. In addition, there was a layer of solidified foam about 1/2-in. thick at the top of the crucible. Calculations showed that the maximum rate of carbon monoxide production prior to clogging was 0.49 scfh (i.e., 22 scfh per sq ft of crucible cross-sectional area). This production velocity should vary with the melt viscosity, type of rock, size of crucible, etc. Prolonged heating of the crucible at temperatures up to 1800°C did not result in full recovery of the carbon; 74% was recovered over a period of 60 hours. The low carbon recovery was caused by the foaming of the carbon and rock out of the crucible (i.e., out of the hot zone). The reduction reaction cannot take place out of the hot zone of the furnace.

Inspection and analyses of the furnace insulation (alumina bubbles) after the run showed the presence of carbon. This carbon could have been deposited by the decomposition of carbon monoxide on the hot insulation after the gas outlet tube clogged.

The spectrographic analyses of the alumina bubbles from Run 7 are presented in Table 4. Figure 5 is a photograph of the bubbles as taken in reverse order from the furnace. Virgin bubbles are composed of nearly pure alumina with about 1.6% silicate as Si. The used bubbles were found to contain more silicon (silica) as their position approached the top of the crucible. The bubbles were also found to be contaminated with appreciable quantities of molybdenum (from the furnace winding), sodium (sublimed from the reduced melt), and lesser quantities of magnesium and iron.

Table 4 presents data on the spectrographic analyses of the metal obtained from Runs 6 and 7. The silicon content of the metal from Run 6 was 62.4%. A material balance shows that the silicon in the metal corresponds to nearly a 50% reduction of the silica in the basalt charged. Sufficient graphite had been added to the crucible to reduce just 50% of the silicate. In Run 7, the silicon content of the metal was only 15.1%; this low content probably came about because the metal found in the bottom of the crucible was formed early in the run (i.e., before the melt foamed over). The metal samples, in addition, contained appreciable quantities of aluminum, manganese, and titanium, and lesser quantities of copper and nickel.

The clogging of the gas outlet tube in Run 7 caused the crucible to swell and adhere to the furnace wall. This necessitated a virtually complete rebuilding of the interior parts of the furnace.

Run 10 was to be a duplicate of Run 6. One pound of basalt mixed with 60.5 g of graphite was used. The basalt and graphite were crushed to the same size as in Run 6. Approximately the same rate of heating was used in Run 10 as in Run 7. However, after about 32 hours of operation, it became apparent that something had gone wrong. First of all, the carbon monoxide content in the product gas began to fall off. After 46 hours of operation, the heating unit shifted in the furnace and broke off the optical pyrometer sight tube. The run was terminated after 47.5 hours of operation with a carbon recovery of only 54.5%. Although some carbon monoxide was still being produced, it was impossible to determine the crucible wall temperature. Inspection of the furnace showed that the melt crucible had broken and allowed some of the molten rock to run into the heating crucible. The reason for failure was not determined.

One positive result obtained from Run 10 was that it was found that the product gases did not contain any sulfur and only a trace of phosphorus. In this run, all of the product gases were bubbled through a 0.1 N NaOH solution. The solution was then analyzed for sulfur and phosphorus. No sulfur and only 1.6 ppm of phosphorus was found. This confirms the results obtained in Run 3.

b. Runs 4, 5, 8, and 9 (Reduction of Silicate with Methane)

Run 4 was made with 1 lb of granite in a 2.5-in.-OD by 8-in.-deep zirconia crucible. Three zirconia inlet bells, 3/4-in. OD by 12-in. long, were used. The upper ends of the bells were joined to 1/8-in.-OD alumina inlet tubes. The joint was made with Zircoa Cast (-100 mesh size). The cams on the timer were also changed so that the melt would be forced into the bells for approximately 2 min out of every 10 min of operation. This was done in an effort to get better removal of the carbon deposited in the bells.

The crucible was heated to 1580°C with an argon purge. The methane (95.0% CH_4, 4.5% H_2, 0.5% N_2) was then turned on at a rate of 0.2 g mole/hour. The crucible temperature was maintained at about 1600°C. The carbon monoxide content of the product gas rose slowly to a maximum of 5.2% after 4.5 hours of methane addition. This value corresponds to a 41% carbon recovery. Thereafter, the carbon monoxide content of the product gas fell slowly; after 11 additional hours, the carbon recovery was only 21%. Appreciable quantities of methane were present in the product gas; the amount gradually increased as the run progressed.

Examination of the furnace and inlet bells after the run showed that the seals between the bells and 1/8-in. inlet tubes were leaking. Apparently, a large part of the methane by-passed the crucible during the run and cracked inside the furnace or escaped unreacted with the product gases.

Run 5 was made with one pound of -6 to +28 mesh basalt in a 2.5-in. OD by 8-in. deep zirconia crucible. Three 3/4-in. OD by 12-in. long zirconia inlet bells were used. The joints between the bells and the 1/8-in. inlet tubes were modified to give a thicker seal of Zircoa Cast than previously used. In addition, the exterior of the joint was covered with several coats of Sauereisen Binder No. 32 (a high-temperature ceramic sealing compound). The same cams on the timer were used as for Run 4, except that an additional valve and timer were added to surge the melt up and down in the bells once a minute for 2 min out of every 10 min of operation.

The crucible was heated to about 1570°C while the system was purged with argon. Carbon monoxide was formed during this warm-up period from the carbon deposited in the furnace insulation during the previous run. Approximately 12 hours of heating at 1570°C was required to eliminate the carbon monoxide from the product gas. During this time, the cycle timers were operated with a low argon flow to test the system and the seals in the inlet tubes and bells. Two of the three bell seals held up perfectly all during this time. The third bell seal opened up almost completely. This was indicated by a large flow in the off-gas rotameter. The methane feed was turned on using the remaining two bells. However, in a short time (1 to 2 hours), one of the bell seals started leaking and the other bell started to clog; the run was then terminated.

Inspection of the bells after the run showed that surging of the melted basalt up and down for 6 hours at about 1570°C had eroded or dissolved more than one-half of the zirconia wall (approximately 0.020 in. penetration). One bell made of a special Zircoa mixture (No. 1027) appeared to hold up no better than the standard impervious zirconia. Figure 6 is a top view of the crucible from Run 5. Small beads of metal can be seen embedded in the top of the solidified melt.

Run 8 was made with 1.0 lb of basalt in a 2-in.-ID, 8-in.-high zirconia crucible. In this run, one-piece inlet tubes made from impervious zirconia were used for the methane inlets. The tubes were approximately 0.52-in. OD, 0.35-in. ID, and 18-in. long. Twisted tungsten wires (three 0.050 in. wires) were installed in two of the three tubes. The third tube, used as a blank, was open in the usual manner. The wire in one of the tubes was rotated at about 60 rpm during the run, while the other wire was raised and lowered a distance of 2 in. about 30 times per minute during the run. The moving wires were used in an attempt to prevent carbon from clogging the gas inlet tubes. The crucible was heated to approximately 1550°C with a low argon purge before the wire stirrers were turned on. The rotating wire became tangled soon after its rotation began; it was then stopped. The reciprocating wire became stuck and inoperable after 2.5 hours. The open tube operated for 10 hours with methane flow before it became clogged with hard carbon.

A 3/8-in.-ID alumina tube was used as the gas outlet from the crucible. A 1/8-in.-OD alumina rod was installed inside of the gas outlet tube to keep it free of sublimate and dust. Raising and lowering the 1/8-in. rod several inches at 15- to 30-min intervals prevented the outlet line from clogging.

Complete data for Run 8 are not reported because of the run's brief duration and low carbon monoxide recoveries. The carbon monoxide recovery rate was 41.6% shortly after the methane was turned on at 1580°C; the rate of carbon monoxide recovery increased to a maximum of 66% approximately 3 hours after the methane was turned on. The carbon monoxide recovery thereafter fell to 36% (13 hours), and to 27% when the run was terminated.

Inspection of the furnace after the run showed that each of the three inlet tubes had carbonized and cracked, allowing the methane to flow into the furnace insulation. This pointed to a need for lining the inlet tubes with some material which will not be penetrated by the carbon in a reducing atmosphere at 1200 to 1600°C. In addition to being cracked, the inlet tubes were filled with hard carbon for about 3 in. starting an inch or two above the crucible top. This served to indicate the need for drilling or reaming the carbon from the tubes as fast as it formed.

Run 9 was made with 1.0 lb of basalt in a 2-in.-ID, 8-in.-high zirconia crucible. In this run, only one 1/2-in.-OD, 18-in.-long zirconia inlet tube was used. The top 15 in. of the zirconia tube was lined with a piece of molybdenum sheet 0.003-in. thick by 2.5-in. wide. The sheet was formed into a spiral shape which was inserted into the tube and expanded to make a reasonably tight inner lining. Three 0.050-in. tungsten wires twisted together were inserted into the inlet tube extending to within 6.5 in. of the bottom of the crucible. After the methane feed was turned on, the tungsten wires were rotated at about 60 rpm for 8 sec out of each minute. The differential pressure surging mechanism was not used during this run.

The crucible was heated at a rate of 800°C/hour with an argon purge up to 1550°C, and then the methane feed was turned on at a rate of 0.20 mole/hour. The wire rotating timer was also turned on at this time. The conversion of methane to CO rose to about 36% shortly after the methane was turned on, and then slowly fell off. No methane was detected in the product gas. After only 2.25 hours, the inlet tube clogged off with carbon stopping the methane flow.

Inspection of the inlet tube (see Figure 7) after the run showed that the tube was totally clogged with carbon about 6.5 to 8.5 in. above the crucible bottom. The tungsten wires had caught and twisted off at a level of about 8 in. above the crucible bottom. The molybdenum liner was effective in preventing the carbon from attacking the zirconia tube. However, the lower 0.5 in. of the molybdenum liner had been corroded away by the melt (to a level of 4.5 in. above the crucible bottom). The movement of a 1/8 in. alumina rod in the product gas outlet tube again prevented this line from clogging with sublimate. These results show that reaming, or some other method, will be required to remove the carbon from the inlet tube.

B. TASK 2, CARBON MONOXIDE REDUCTION, METHANE SYNTHESIS

1. Reactor Modifications*

Only one modification was made to the carbon monoxide reduction reactor during this report period. This modification involved placing a sensitive needle valve in the upstream (gas) leg of the product water dumping manometer. This was done to suppress the surging of the mercury column whenever the solenoid valve was actuated. This surging in the past had resulted in occasional loss of gas as well as water. This loss of gas actuated the low-pressure shutdown switch, thereby shutting down the reactor.

2. Reduction Experiments

The second long-duration carbon monoxide reduction run was started during this report period. A continuous 90-day test at a space velocity of 1000 hr^{-1} is planned. The catalyst is the same as that used in the first long-duration run. The reactor had operated for 1284 hours (53.5 days) with little indication of change in catalyst performance. The data for this test are reported in Tables 5 through 7. The conditions and product yields for Run 2 are listed in Tables 5 and 7; the product gas analyses are given in Table 6. The carbon monoxide conversion remains at 100%.

At the beginning of the run the carbon dioxide yield was relatively high, but dropped to a nominal level after approximately 12 hours, when equilibrium conditions were reached. The average percent yields of products for Run 2 is as follows: methane, 102.6; water, 99.9; and carbon dioxide, 0.10. The carbon material balance is 102.7, while the oxygen material balance is 100.1. These results are within the limits of error of the instrumentation and analytical methods. This 90-day run is being continued.

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*See Aerojet-General Quarterly Reports 0765-03-1 and -2, Contract NAS 7-225 (October 1964 and January 1965, respectively) for a complete description of the silicate reduction unit.

IV. FUTURE WORK

A. TASK 1, CARBOTHERMAL REDUCTION OF SILICATES

Reduction of silicates with methane will continue with the use of a rotating reaming device fabricated with tungsten carbide to remove carbon deposited in the reactant gas inlet tube.

B. TASK 2, CARBON MONOXIDE REDUCTION, METHANE SYNTHESIS

The 90-day run will be completed.

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 975 man-hours and $14,200 were expended on Contract NAS 7-225.

TABLE 1

SUMMARY OF SILICATE REDUCTION RUNS

Run No.	Material Charged	Methane Feed Rate (mole/hr)	Inlet Tube Description	Outlet Tube Description	Run Duration (hr)	Maximum Conversion (%)	Comments
4	454 g graphite -6 to +28 mesh	0.20	Three 3/4-in. OD x 12-in. long ZrO_2 bells with zircoa cast seals; surging	1/4-in. ID Al_2O_3	21	41	Bells leaked; C deposited in bells.
5	454 g basalt -6 to +28 mesh	0.20	Three 3/4-in. OD x 12-in. long ZrO_2 bells with zircoa and Sauereisen seals; surging	1/4-in. ID Al_2O_3	2	70	Two bells leaked; one bell clogged.
8	454 g basalt -6 to +28 mesh	0.20	Three 1/2-in. ID x 18-in. long ZrO_2 tubes; tungsten wires moving; no surging	3/8-in. ID Al_2O_3 1/8-in. OD rod	16	66	One tube clogged; two tubes broke.
9	454 g basalt -6 to +28	0.20	One 1/2-in. OD x 18-in. long ZrO_2 tube; tungsten wire rotating; Mo tube liner; no surging	3/8-in. ID Al_2O_3 1/8-in. OD rod	2.25	36	Wire broke; tube clogged.
6	454 g basalt plus 60.5 g graphite -6 to +28 mesh	0	None	1/2-in. ID Al_2O_3	68	86	Slow reaction; outlet line clogged.
7	454 g basalt plus 60.5 g graphite -28 to +48 mesh	0	None	1/2-in. ID Al_2O_3	60	74	Melt foamed and clogged outlet.
10	454 g basalt plus 60.5 g graphite -6 to +28 mesh	0	None	1/2-in. ID Al_2O_3 1/8-in. OD rod	47.5	54.5	Crucible cracked.

TABLE 2

REDUCTION OF BASALT WITH GRAPHITE*

RUN 6

Time (hours)	Crucible Temp (°C)	Product Gas Rate (gram mole/hour)	Carbon Monoxide in Product Gas (mole %)	Carbon Recovered as CO (g carbon)	Cumulative** Recovery of Carbon (%)
0	1020	1.32	1.4	0	0
4	1310	1.31	4.6	2.56	4.2
8	1400	1.25	7.6	6.71	11.1
12	1560	1.26	10.3	11.30	18.7
16	1560	1.16	4.3	15.26	25.2
20	1640	1.28	10.4	19.72	32.6
24	1580	1.21	7.0	23.26	38.4
28	1615	1.23	7.1	26.67	44.1
32	1500	0	0	30.98	51.1
36	1590	1.14	0.3	31.02	51.3
40	1690	1.28	8.8	34.13	56.4
44	1690	1.19	6.7	39.32	65.0
48	1690	1.12	7.0	44.27	73.2
52	1680	1.07	4.0	46.82	77.4
56	1685	1.04	3.0	48.54	80.1
60	1700	1.05	2.3	49.82	82.4
64	1730	1.05	2.4	51.05	84.3
68	1785	1.10	2.0	52.13	86.1

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[*] 454 g of basalt mixed with 60.5 g of pure graphite in an impervious zirconia crucible and heated with an argon purge.

[**] In addition to the CO in the product gas, 0.54 g of H_2O and 0.15 g of CO_2 were recovered.

TABLE 3

REDUCTION OF BASALT WITH GRAPHITE*

RUN 7

Time (hours)	Crucible Temp (°C)	Carbon Monoxide in Product Gas (mole %)	Carbon Recovered as CO (g carbon)	Cumulative** Recovery of Carbon (%)
0	660	0	0	0
2	1070	4.2	0.37	0.6
4	1305	7.8	2.82	4.7
6	1445	7.4	4.85	8.0
8	1562	16.7	10.23	16.9
10	1575	30.8	17.35	28.7
12	1592	5.7	22.21	36.8
14	1658	5.4	23.66	39.1
16	1685	8.0	25.38	42.0
18	1715	6.0	27.15	44.9
20	1710	4.3	28.48	47.1
22	1705	2.2	29.27	48.2
24	1700	1.5	29.69	49.1
26	1725	1.8	30.09	49.7
28	1755	5.7	31.30	51.7
30	1755	4.4	32.61	53.8
32	1755	3.7	33.59	55.5
34	1755	3.6	34.54	57.1
36	1745	3.2	35.37	58.4
38	1750	3.3	36.18	59.8
40	1758	3.2	37.00	61.2
42	1755	3.6	37.95	62.8
44	1745	3.0	38.77	64.1
46	1760	2.8	39.49	65.2
48	1765	2.6	40.16	66.3
50	1782	2.9	40.87	67.7
52	1790	4.5	41.90	69.2
54	1765	3.0	42.86	70.9
56	1758	2.4	43.75	72.5
58	1765	2.2	44.32	73.4
60	1758	1.8	44.78	74.1

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* 454 g of basalt mixed with 60.5 g of pure graphite in an impervious zirconia crucible. The graphite was crushed to -28 mesh to +40 mesh.

** In addition to the CO in the product gas, 1.78 g of H_2O and 0.21 g of CO_2 were recovered.

TABLE 4

EMISSION SPECTROGRAPHIC ANALYSES

	Sample Analysis (Wt%)					
			Alumina Bubbles from Run 7			
Component	Metal from Run 6	Metal from Run 7	New Bubbles	1/4 way down to Crucible**	3/4 way Down to Crucible	Near Crucible Top
Si	62.4	15.1	1.6	7.8	6.9	15.1
Fe	23.5	75.4	-	1.1	0.2	0.2
Mg	0.8	0.1	-	1.0	2.4	2.3
Al	5.8	1.4	98.4	73.5	72.0	65.7
Cu	0.4	0.6	-	-	-	-
Mn	5.8	2.3	-	-	-	-
Ti	1.3	2.0	-	-	-	-
Ni	-	1.4	-	-	-	-
Mo	-	-	-	8.8	14.7	11.7
Na	-	-	-	7.8	3.8	5.0
Co	-	1.7	-	-	-	-
C*	-	-	-	-	0.2	-

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* Carbon analyses by furnace oxidation to CO_2 and subsequent absorption

** From top of insulation layer

TABLE 5

REDUCTION OF CARBON MONOXIDE WITH HYDROGEN

(Operating Data)

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)	Catalyst Bed Static (psig)	Catalyst Bed ΔP (in.Hg)	Water Jacket (°C)	Catalyst Bed Maximum[a] (°C)
2-1	0-24	3.81	1.23	1.36	21.6	615	75.5	0.14	255	345
2-2	24-66	3.81	1.23	1.38	21.7	600	75.5	0.14	254	344
2-3	66-89	3.81	1.23	1.37	21.6	555	76.0	0.14	249	343
2-4	89-127	3.81	1.23	1.36	21.9	585	76.0	0.14	253	346
2-5	127-144	3.81	1.23	1.39	22.1	570	75.5	0.14	251	350
2-6	144-168	3.81	1.23	1.36	22.0	570	76.0	0.14	250	348
2-7	168-190	3.81	1.23	1.36	20.8	565	76.0	0.14	250	348
2-8	190-214	3.81	1.23	1.37	21.9	565	76.0	0.14	250	348

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

TABLE 5 (cont.)

REDUCTION OF CARBON MONOXIDE WITH HYDROGEN

(Operating Data)

		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[a] (°C)
2-9	214-239	3.81	1.23	1.35	22.4	575	75.5	0.15	251	346
2-10	239-262	3.81	1.23	1.37	22.2	555	76.0	0.15	250	349
2-11	262-280	3.81	1.23	1.35	22.1	555	76.0	0.15	250	352
2-12	280-304	3.81	1.23	1.33	22.4	555	76.0	0.15	250	352
2-13	304-327	3.81	1.23	1.36	22.0	560	76.0	0.15	250	350
2-14	327-351	3.81	1.23	1.36	22.0	560	76.0	0.15	250	348
2-15	351-375	3.81	1.23	1.35	22.3	550	76.0	0.15	249	347
2-16	375-400	3.81	1.23	1.36	22.0	550	76.0	0.16	249	347
2-17	400-423	3.81	1.23	1.35	22.1	570	76.0	0.16	251	349
2-18	423-456	3.81	1.23	1.41	22.2	575	76.0	0.16	252	350
2-19	456-495	3.81	1.23	1.41	22.3	565	76.5	0.16	252	350
2-20	495-519	3.81	1.23	1.43	22.3	565	76.5	0.16	252	349
2-21	519-543	3.81	1.23	1.43	22.4	560	76.5	0.16	252	348
2-22	543-574	3.81	1.23	1.40	22.1	560	76.5	0.16	251	346
2-23	574-599	3.81	1.23	1.42	22.1	560	76.0	0.16	250	345
2-24	599-623	3.81	1.23	1.42	22.3	565	76.0	0.16	251	347
2-25	623-664	3.81	1.23	1.43	22.4	570	76.5	0.16	252	349
2-26	664-688	3.81	1.23	1.44	22.1	565	76.0	0.16	252	349
2-27	688-732	3.81	1.23	1.43	22.0	560	76.0	0.16	252	349
2-28	732-766	3.81	1.23	1.42	22.2	560	76.5	0.16	252	349
2-29	766-786	3.81	1.23	1.43	22.2	570	76.5	0.16	252	349
2-30	786-816	3.81	1.23	1.41	22.3	585	76.5	0.16	253	350
2-31	816-831	3.81	1.23	1.42	21.9	575	76.5	0.16	253	350

TABLE 5 (cont.)

REDUCTION OF CARBON MONOXIDE WITH HYDROGEN

(Operating Data)

		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 (°C)
2-32	831-855	3.81	1.23	1.41	22.4	560	76.5	0.16	252	345
2-33	855-878	3.81	1.23	1.39	21.9	560	76.5	0.16	251	341
2-34	878-931	3.81	1.23	1.39	22.3	560	76.5	0.16	251	341
2-35	931-979	3.81	1.23	1.41	22.1	570	76.5	0.16	252	341
2-36	979-997	3.81	1.23	1.44	22.3	565	76.5	0.16	251	342
2-37	997-1029	3.81	1.23	1.42	22.4	550	76.5	0.16	252	340
2-38	1029-1048	3.81	1.23	1.42	22.1	550	76.5	0.16	254	339
2-39	1048-1072	3.81	1.23	1.44	22.2	555	76.5	0.16	253	341
2-40	1072-1097	3.81	1.23	1.45	22.3	565	76.0	0.16	251	335
2-41	1097-1118	3.81	1.23	1.48	22.6	570	76.0	0.16	253	334
2-42	1118-1140	3.81	1.23	1.46	22.4	580	76.5	0.16	254	335
2-43	1140-1183	3.81	1.23	1.49	22.5	575	76.5	0.16	252	333
2-44	1183-1213	3.81	1.23	1.52	22.2	555	76.5	0.15	251	331
2-45	1213-1236	3.81	1.23	1.48	22.5	550	76.5	0.15	251	331
-46	1236-1284	3.81	1.23	1.51	22.3	545	76.5	0.15	250	330

TABLE 6

PRODUCT GAS ANALYSIS

Run No.	Composition of Product Gas (Vol %)				
	H_2	CO	CH_4	CO_2	H_2O
2-1	8.5	0.0	90.5	0.25	0.4
2-2	8.4	0.0	90.8	0.10	0.4
2-3	8.7	0.0	90.6	0.05	0.4
2-4	7.0	0.0	92.0	0.15	0.4
2-5	8.9	0.0	89.8	0.10	0.4
2-6	8.0	0.0	90.6	0.10	0.4
2-7	7.2	0.0	91.4	0.10	0.4
2-8	7.0	0.0	91.5	0.15	0.4

TABLE 6 (cont.)

PRODUCT GAS ANALYSIS

Run No.	Composition of Product Gas (Vol %)				
	H_2	CO	CH_4	CO_2	H_2O
2-9	6.3	0.0	92.7	0.20	0.4
2-10	6.6	0.0	92.4	0.35	0.4
2-11	6.8	0.0	92.2	0.35	0.4
2-12	7.3	0.0	91.7	0.30	0.4
2-13	8.1	0.0	90.8	0.10	0.4
2-14	8.4	0.0	90.2	0.10	0.4
2-15	6.6	0.0	92.2	0.15	0.4
2-16	5.0	0.0	93.9	0.40	0.4
2-17	5.6	0.0	93.2	0.45	0.4
2-18	7.4	0.0	91.7	0.15	0.4
2-19	8.9	0.0	90.3	0.05	0.4
2-20	9.4	0.0	89.7	0.0	0.4
2-21	9.5	0.0	89.6	0.0	0.4
2-22	8.7	0.0	90.3	0.0	0.4
2-23	9.3	0.0	89.9	0.0	0.4
2-24	9.7	0.0	89.5	0.05	0.4
2-25	10.0	0.0	89.1	0.05	0.4
2-26	10.9	0.0	88.3	0.0	0.4
2-27	10.5	0.0	88.7	0.0	0.4
2-28	9.9	0.0	89.3	0.0	0.4
2-29	10.2	0.0	89.0	0.0	0.4
2-30	10.2	0.0	89.0	0.0	0.4
2-31	10.0	0.0	89.2	0.0	0.4
2-32	10.7	0.0	88.5	0.05	0.4
2-33	10.1	0.0	89.1	0.10	0.4
2-34	8.6	0.0	90.6	0.10	0.4
2-35	8.8	0.0	90.5	0.0	0.4
2-36	10.2	0.0	89.1	0.0	0.4
2-37	10.0	0.0	89.2	0.0	0.4
2-38	9.1	0.0	90.1	0.0	0.4
2-39	10.6	0.0	88.6	0.0	0.4
2-40	11.8	0.0	86.8	0.55	0.4
2-41	11.5	0.0	87.8	0.0	0.4
2-42	11.2	0.0	88.1	0.0	0.4
2-43	12.4	0.0	87.0	0.0	0.4
2-44	13.5	0.0	85.8	0.05	0.4
2-45	13.4	0.0	85.9	0.05	0.4
2-46	13.7	0.0	85.7	0.05	0.4

TABLE 7

CARBON MONOXIDE CONVERSIONS AND PRODUCT YIELDS

Run No.	Nominal H_2/CO Mole Ratio	Nominal Space Velocity (hr^{-1})	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		
					H_2O	CH_4	CO_2
2-1	3.1:1	1000	250	100	97.8	100.0	0.25
2-2	3.1:1	1000	250	100	98.2	101.5	0.10
2-3	3.1:1	1000	250	100	97.8	101.1	0.05
2-4	3.1:1	1000	250	100	99.1	101.8	0.15
2-5	3.1:1	1000	250	100	100.3	101.7	0.10
2-6	3.1:1	1000	250	100	99.6	100.4	0.10
2-7	3.1:1	1000	250	100	94.5	100.8	0.15
2-8	3.1:1	1000	250	100	99.4	101.6	0.20

TABLE 7 (cont.)

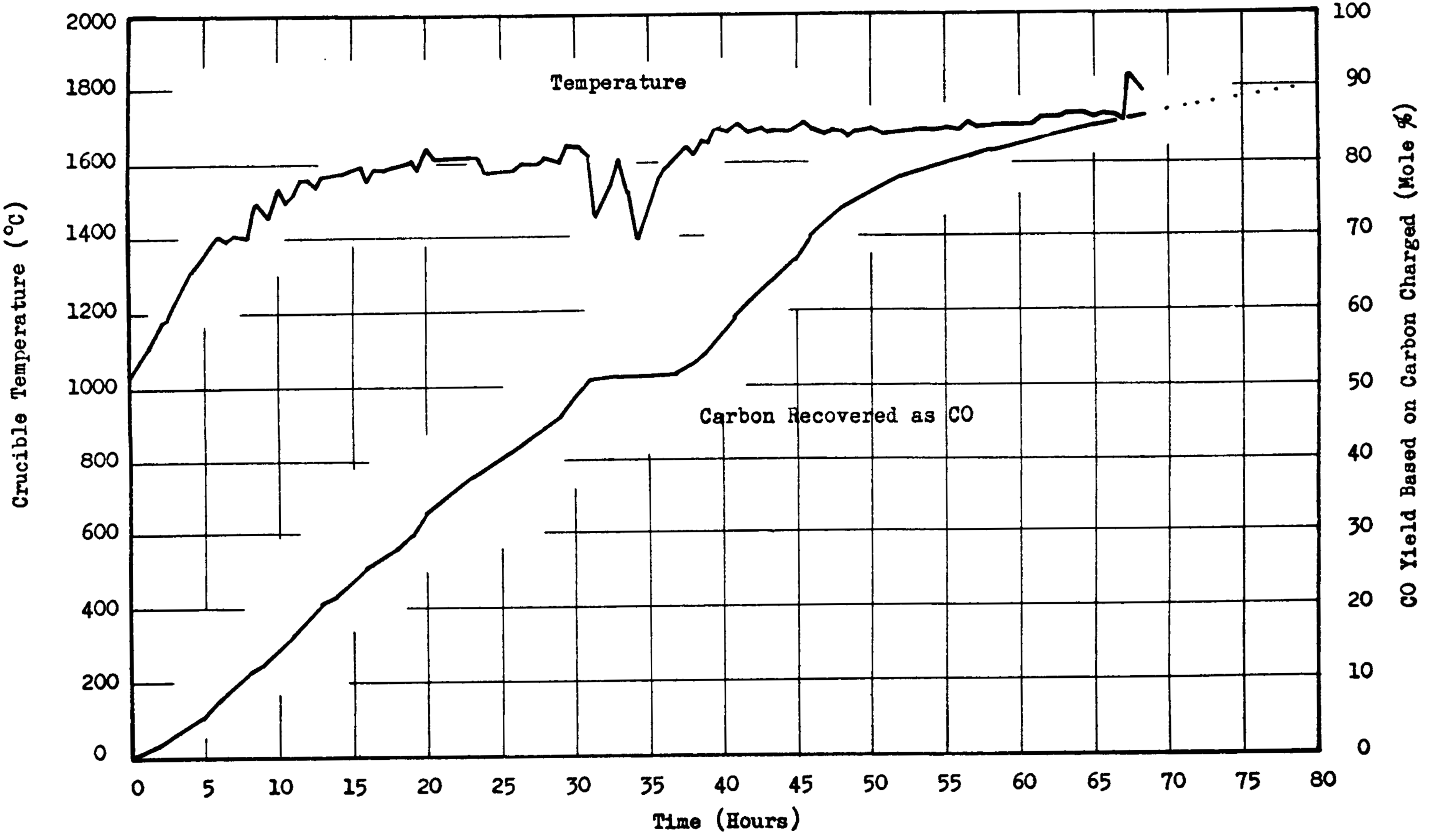
CARBON MONOXIDE CONVERSIONS AND PRODUCT YIELDS

Run No.	Nominal H_2/CO Mole Ratio	Nominal Space Velocity (hr^{-1})	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		
					H_2O	CH_4	CO_2
2-9	3.1:1	1000	250	100	100.9	101.7	0.25
2-10	3.1:1	1000	250	100	100.3	102.9	0.35
2-11	3.1:1	1000	250	100	99.7	101.2	0.40
2-12	3.1:1	1000	250	100	101.4	101.8	0.30
2-13	3.1:1	1000	250	100	99.3	100.3	0.10
2-14	3.1:1	1000	250	100	99.1	100.1	0.10
2-15	3.1:1	1000	250	100	99.8	100.6	0.15
2-16	3.1:1	1000	250	100	99.1	103.4	0.45
2-17	3.1:1	1000	250	100	99.7	102.4	0.50
2-18	3.1:1	1000	250	100	100.3	105.2	0.20
2-19	3.1:1	1000	250	100	100.5	103.8	0.05
2-20	3.1:1	1000	250	100	100.6	104.4	0.00
2-21	3.1:1	1000	250	100	101.2	104.3	0.00
2-22	3.1:1	1000	250	100	100.2	103.1	0.00
2-23	3.1:1	1000	250	100	99.7	104.0	0.00
2-24	3.1:1	1000	250	100	100.2	103.3	0.05
2-25	3.1:1	1000	250	100	100.2	103.1	0.05
2-26	3.1:1	1000	250	100	99.6	103.1	0.00
2-27	3.1:1	1000	250	100	99.3	103.0	0.00
2-28	3.1:1	1000	250	100	100.2	103.3	0.00
2-29	3.1:1	1000	250	100	100.3	103.8	0.00
2-30	3.1:1	1000	250	100	100.7	101.9	0.00
2-31	3.1:1	1000	250	100	98.7	102.7	0.00

TABLE 7 (cont.)

CARBON MONOXIDE CONVERSIONS AND PRODUCT YIELDS

Run No.	Nominal H_2/CO Mole Ratio	Nominal Space Velocity (hr^{-1})	Nominal Catalyst Bed Temp (°C)	CO Conversion (mole %)	Actual Product Yield (mole %)		
					H_2O	CH_4	CO_2
2-32	3.1:1	1000	250	100	100.9	101.8	0.05
2-33	3.1:1	1000	250	100	99.0	100.9	0.10
2-34	3.1:1	1000	250	100	100.4	102.1	0.10
2-35	3.1:1	1000	250	100	99.8	103.7	0.00
2-36	3.1:1	1000	250	100	100.7	103.8	0.00
2-37	3.1:1	1000	250	100	101.2	103.2	0.00
2-38	3.1:1	1000	250	100	99.9	104.4	0.00
2-39	3.1:1	1000	250	100	100.0	103.7	0.00
2-40	3.1:1	1000	250	100	100.7	102.4	0.65
2-41	3.1:1	1000	250	100	102.2	105.8	0.00
2-42	3.1:1	1000	250	100	100.9	104.6	0.00
2-43	3.1:1	1000	250	100	101.5	105.4	0.00
2-44	3.1:1	1000	250	100	100.4	105.8	0.05
2-45	3.1:1	1000	250	100	101.3	103.1	0.10
2-46	3.1:1	1000	250	100	100.4	105.2	0.05
				Average	99.9	102.6	0.10

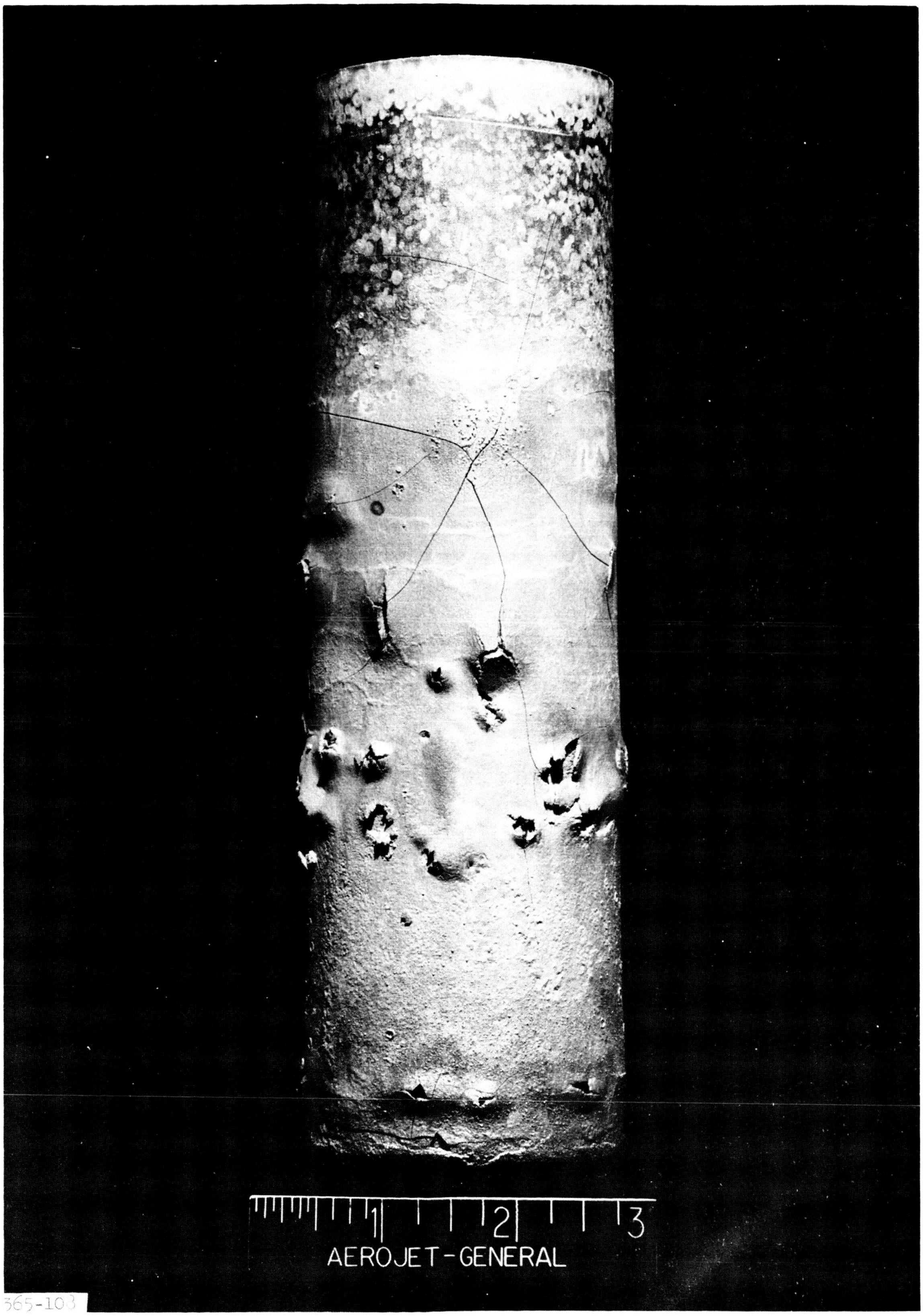


LOG OF RUN 6, REDUCTION OF BASALT WITH CARBON

Figure 1

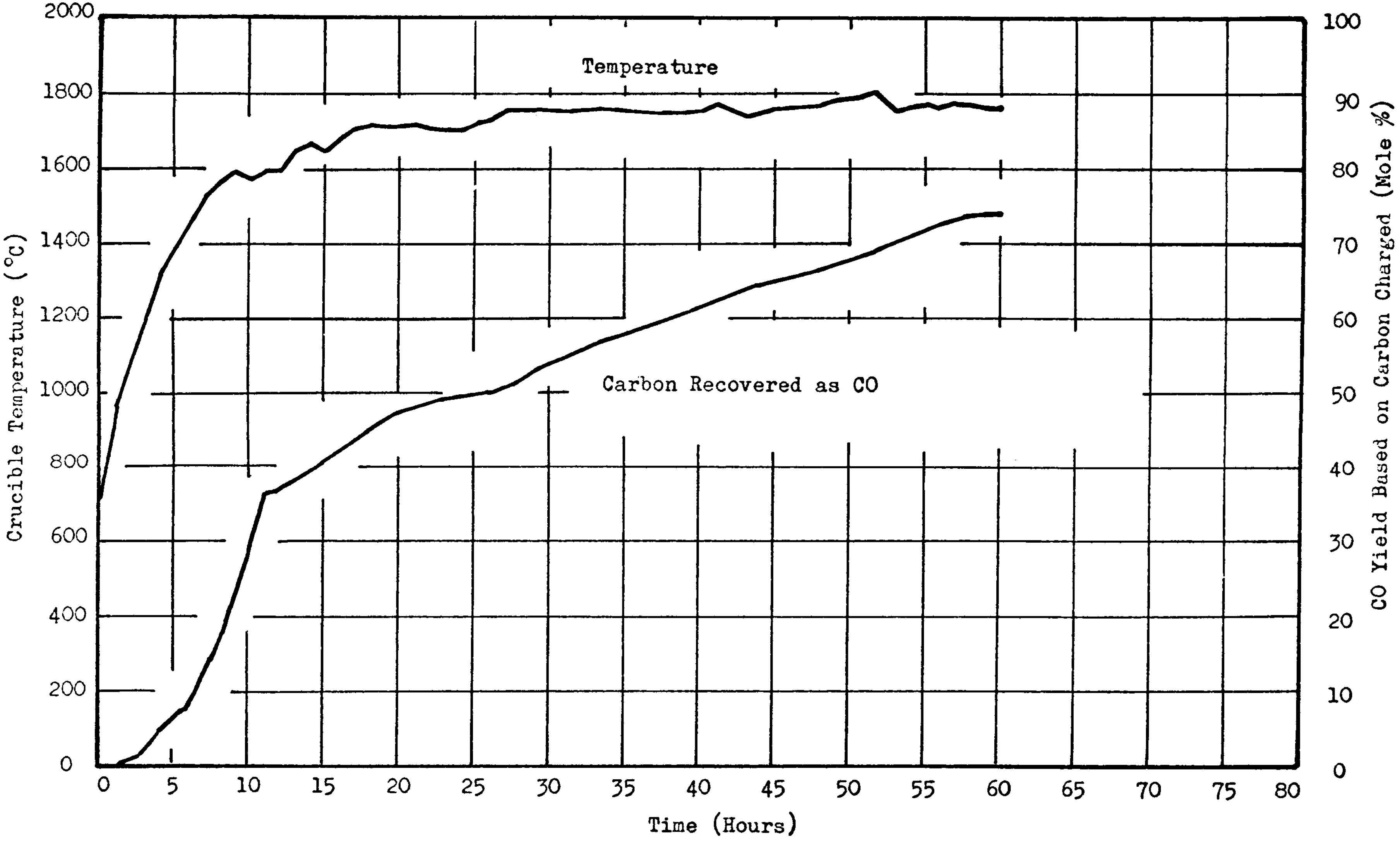
Inside View of Zirconia Curcible from Run 6
(large metal slug at bottom of crucible showed the following analysis:
Si, 62.4; Fe, 23.5; Al, 5.8; Mn, 5.8; Ti, 1.3; Mg, 0.8; and Cu, 0.4)

Figure 2



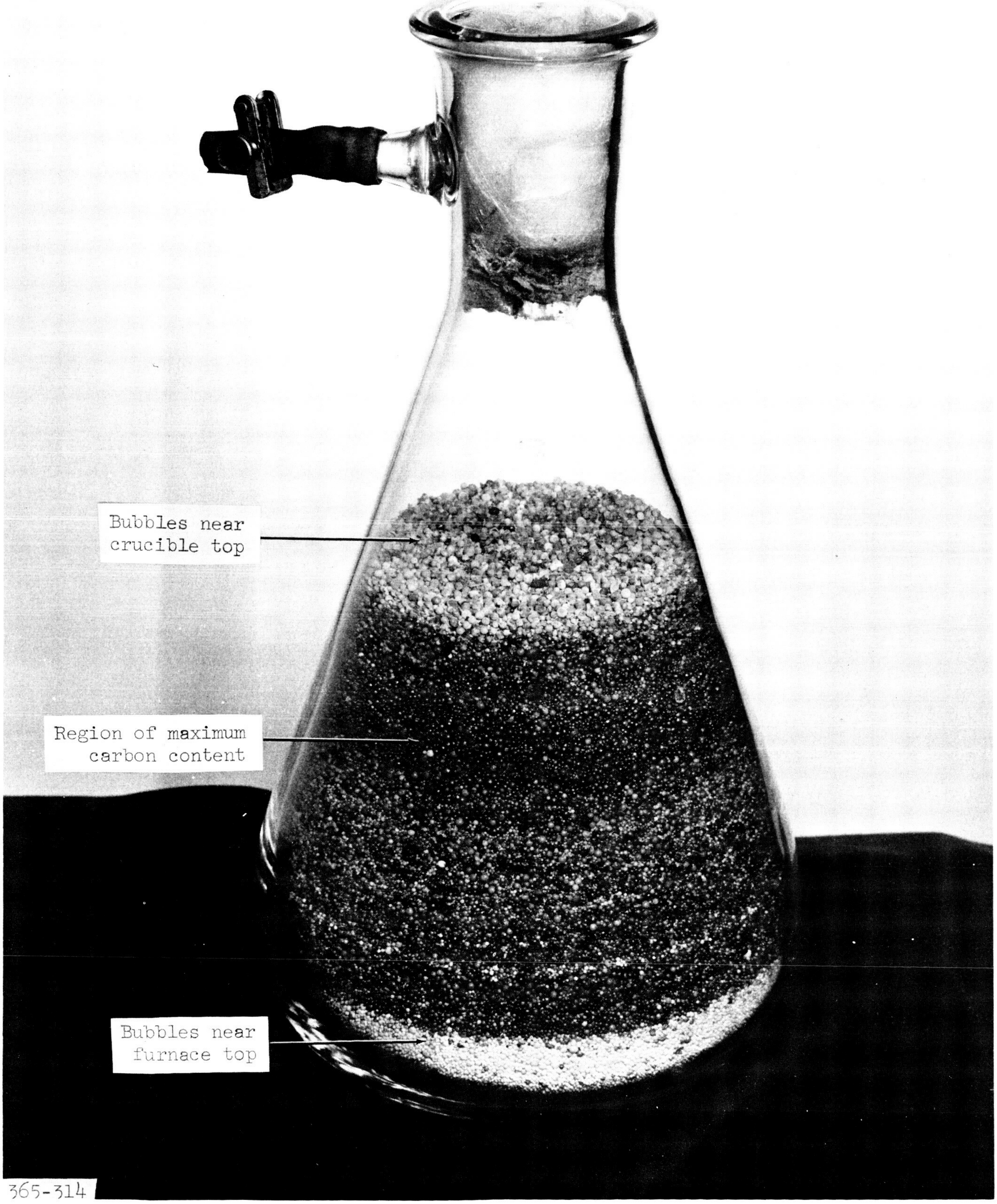
Zirconia Crucible from Run 6 - Outside View

Figure 3



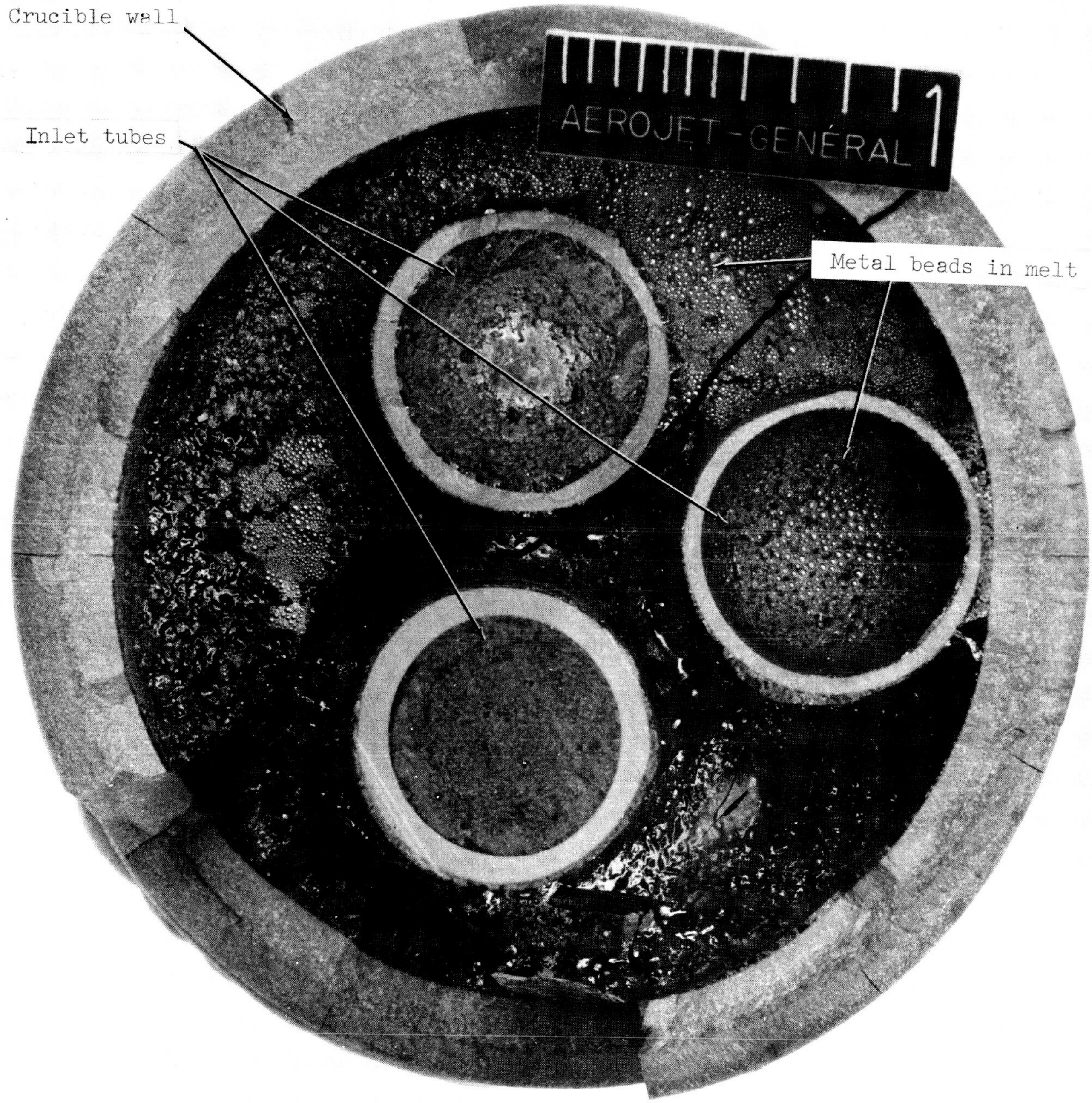
LOG OF RUN 7, REDUCTION OF BASALT WITH CARBON

Figure 4



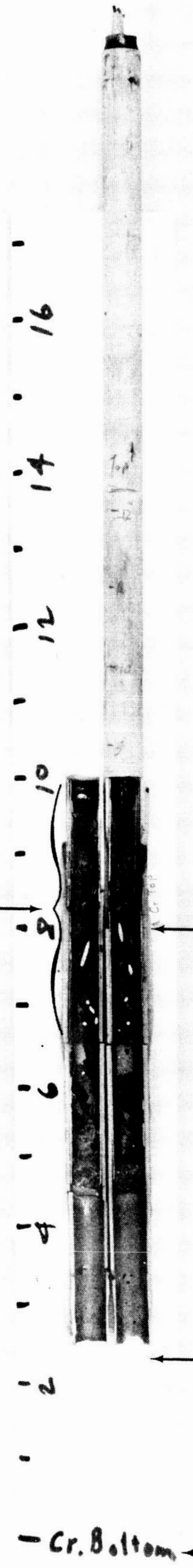
Alumina Bubble Furnace Insulation from Run 7

Figure 5



265-405

Zirconia Crucible from Run 5 - Top View



Region of maximum carbon deposition

Crucible top

Top of melt

Crucible bottom

265-0050

Reactant Gas Inlet Tube from Run 9

Figure 7

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