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UNITED STATES
DEPARTMENT OF THE INTERIOR
J. A. KRUG, SECRETARY

BUREAU OF MINES
R. R. SAYERS, DIRECTOR

REPORT OF INVESTIGATIONS

SPONGE-IRON EXPERIMENTS AT LONGVIEW, TEX.



BY

WHITMAN E. BROWN

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By Whitman E. Brown^{2/}

CONTENTS

	<u>Page</u>
Introduction.....	3
Acknowledgments.....	4
Plant and process.....	4
Description of plant.....	5
Air and gas mixer.....	5
Gas generator and catalyst.....	6
Surge tank and burner.....	6
Ore reducers.....	7
Sponge iron storage cars.....	7
Control room.....	7
Investigations of the process.....	8
Gas re-forming.....	9
Some operating difficulties.....	17
Thermal reactions.....	20
Conclusions on re-forming of gas.....	24
Ore-reduction program.....	24
Beneficiation tests.....	24
Use of coke for improving quality of the gas.....	39
Reduction reactions.....	41
Significance of data and observation with respect to established facts..	48
Factors affecting operation not definitely evaluated.....	50
Changes in plant design to allow commercial or semicommercial operation.	51
Estimate of capital and operating costs for small commercial plant (capacity, 100 tons a day).....	53
Electric-furnace melting of sponge iron.....	55
Summary of melting tests.....	57
Conclusions.....	58

^{1/} The Bureau of Mines will welcome reprinting of this paper provided the following footnote acknowledgment is used: "Reprinted from Bureau of Mines Report of Investigations 3925."

^{2/} Metallurgist, Bureau of Mines, U. S. Department of the Interior.

ILLUSTRATIONS

<u>Fig.</u>		<u>Following page</u>
1.	Schematic diagram of sponge-iron plant at Longview, Tex.	4
2.	Sponge-iron plant, Longview, Tex.	4
3.	Pulsating-valve cycles.....	4
4.	Gas re-former, showing inside construction.....	6
5.	Surge-tank burner.....	6
6.	Surge-tank burner in place.....	4
7.	Sketch of ore reduction chamber, showing position of thermocouples.....	6
8.	Lower reducer-bottom construction.....	6
9.	Sponge-discharging operations.....	6
10.	Sponge-iron storage cars.....	6
11.	Percent reducing gases vs. air:gas ratio.....	18
12.	Percent reducing gases vs. generator pressure.....	18
13.	Percent reducing gases vs. air:gas ratio.....	18
14.	Reducing gas vs. air flow.....	18
15.	Reduction reactions, test 9.....	32
16.	Large lump of reduced ore.....	36
17.	Hydrogen and carbon monoxide equilibria with iron and iron oxides.....	42
18.	Equilibrium constants.....	42
19.	Reduction test 26.....	46
20.	Spent-gas composition vs. reduction time.....	46
21.	Spent-gas composition vs. reduction time.....	46
22.	Spent-gas composition vs. reduction time.....	46
23.	Reaction heats vs. preheat of reagents.....	46
24.	Reaction heats vs. preheat of reagents.....	46
25.	Schematic diagram for rebuilt Longview plant.....	52
26.	Some ingots and casting made from sponge iron melted in the electric furnace.....	36

TABLES

<u>Table</u>		<u>Page</u>
1.	Heat in products.....	22
2.	Roasting and magnetic separation test, minus 100-mesh ore.....	25
3.	Summary of data on tests for production of sponge iron at Longview, Tex.	27
4.	Gas from combustion of several air-natural gas mixtures, percent (dry basis).....	37
5.	Gas obtained after passing combustion gases through incandescent coke, percent (dry basis).....	37
6.	Gas obtained after passing both generator gas and combustion gas through incandescent coke, percent (dry basis).....	38
7.	Comparison of gas mixture not passed through coke and same gas mixture passed through coke.....	38
8.	Effect of mixing generator gas with combustion gas and not passing through incandescent coke.....	39

INTRODUCTION

As part of a Nation-wide program of the United States Government to develop natural resources, funds were made available to the Bureau of Mines for the investigation of the "Reduction of Iron Ores by Solid and Gaseous Fuels." These investigations are concerned with the direct production of iron from its ores without the use of a blast furnace. Many processes have been proposed for the low-temperature reduction of iron oxides, but few have been developed to commercial use. High-quality sponge iron is produced commercially only in Sweden.

The cost of iron production depends in part on the grade of the ore and increases with decrease in grade. The substitution of cheap natural gas in a sponge-iron process for expensive coke in smelting in the blast furnace would partly compensate for the use of low-grade ores. If a process can be developed in which inexpensive natural gas can be used as fuel and reducing agent, the large deposits of low-grade iron ore and the natural-gas resources of east Texas may be utilized in the commercial production of sponge iron.

Sponge iron may possibly be used to supplement scrap iron, pig iron, or, in special instances, wrought iron. Sponge iron will vary but slightly in composition and will contain little or no nickel, cobalt, manganese, copper, and other alloying metals. It is becoming increasingly difficult to obtain choice scrap iron of nonalloy composition. Pig iron contains 1.5 to 3.0 percent silicon and 3.0 to 4.5 percent carbon. Sponge iron generally contains less carbon than pig iron.

The Madaras Steel Corp. in Longview, Tex., built a semicommercial-scale sponge-iron plant which was intended to be operated in conjunction with a steel foundry. The process is based upon the reduction of preheated iron ore with hot reducing gases under pulsating pressure. Although the Madaras Corp. had previously experimented with the production of sponge iron by reducing the ore with pure hydrogen, several attempts to use a re-formed natural gas as the reducing agent in this pilot plant were unsuccessful. The Bureau of Mines leased this plant and conducted an extensive investigation of the Madaras process on a semicommercial scale.

The experiments were not entirely satisfactory, partly because of mechanical difficulties and partly because the reducing gas was not of the best quality. However, much information on the production of reducing gas by re-forming natural gas and on the reduction of iron ore with re-formed natural gas was obtained.

The sponge iron produced contained approximately 0.1 percent phosphorus; and, after melting in an electric furnace, the iron contained approximately 0.15 percent phosphorus. The results of attempts to improve the ore by ore-dressing methods were not encouraging.

ACKNOWLEDGMENTS

This paper is one of many reporting on various aspects of the Bureau of Mines program directed toward the more effective utilization of our mineral resources.

Investigations of our mineral resources are carried out by the Mining Branch, L. B. Moon, Chief, and the Metallurgical Branch, R. G. Knickerbocker, Chief. Both branches are under the supervision of R. S. Dean, Assistant Director.

The scope of this paper falls in the province of the Metallurgical Branch, whose activities embrace the separation of difficulty beneficiated ores, the production of pure metals from domestic deposits, the exploitation of marginal ore reserves, the recovery of secondary metals, and the improvement of present industrial metallurgical practice.

With respect to this report, special acknowledgment is due E. P. Barrett of the Minneapolis Station for extensive review of the report and to C. Travis Anderson of the Rolla Station for valuable suggestions during the experimental program and in preparing manuscript. Acknowledgment also is made to Mathew M. Rose, reduction-plant engineer, Madaras Steel Corp. of Texas, and Grady Shipp, manager, Chamber of Commerce, Longview, Tex.

PLANT AND PROCESS

A schematic diagram of the plant is shown in figure 1 and a photograph in figure 2. In the Madaras process, heated reducing gas is passed through a stationary bed of preheated ore. The reducing gas enters and leaves the reaction chamber or reducer in pulsations caused by the operation of two quick-opening valves, one on the inlet and one on the outlet of the reducer. As the outlet or upper valve of the reducer is closed, the lower or inlet valve is opened, and the reducing gas rushes in until about 35 pounds pressure is developed. At this pressure the inlet valve closes, the outlet valve opens, and the spent gas is expelled to the atmosphere. The relative positions of the pulsating valves are shown in figure 1. Figure 3 shows the operating cycles of the pulsating valves.

Three principal reasons were involved in the choice of pulsating of flow: (1) The building up of pressure would cause deeper and more effective penetration of the gas into the ore particles; moreover, increased pressure increases the quantity of reacting gas in a given volume, thus increasing the rate of reaction. (2) Channeling of the gas stream would be eliminated. (3) Reduction of pressure would result in quick removal of most of the partly spent gas.

The reduction of iron oxides by methane and ethane is very slow, and the reactions are highly endothermic. To provide a more reactive gas, carbon monoxide and hydrogen were produced by partial combustion of the hydrocarbons in the natural gas.

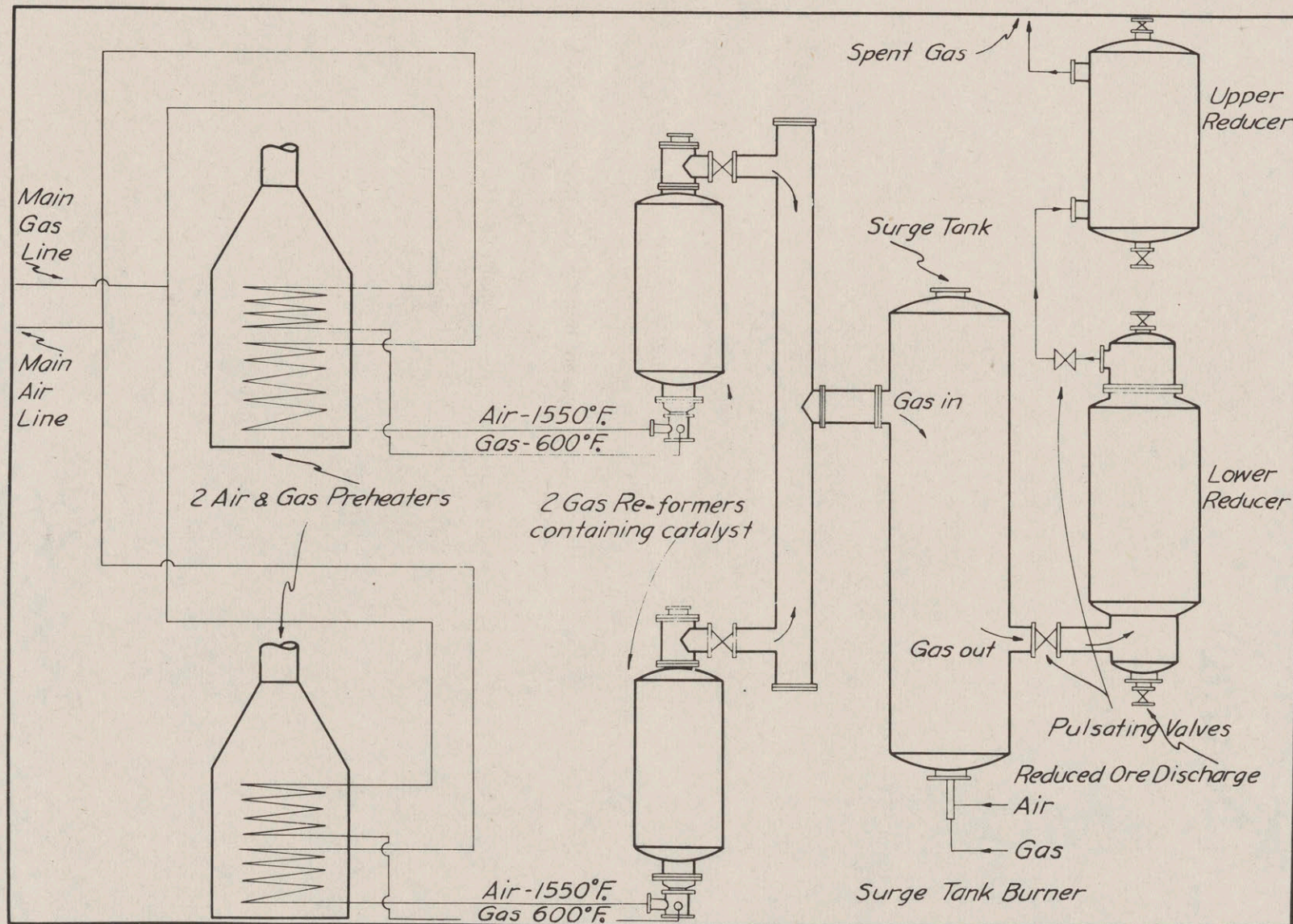


FIGURE 1.- Schematic diagram of sponge-iron plant at Longview, Tex.

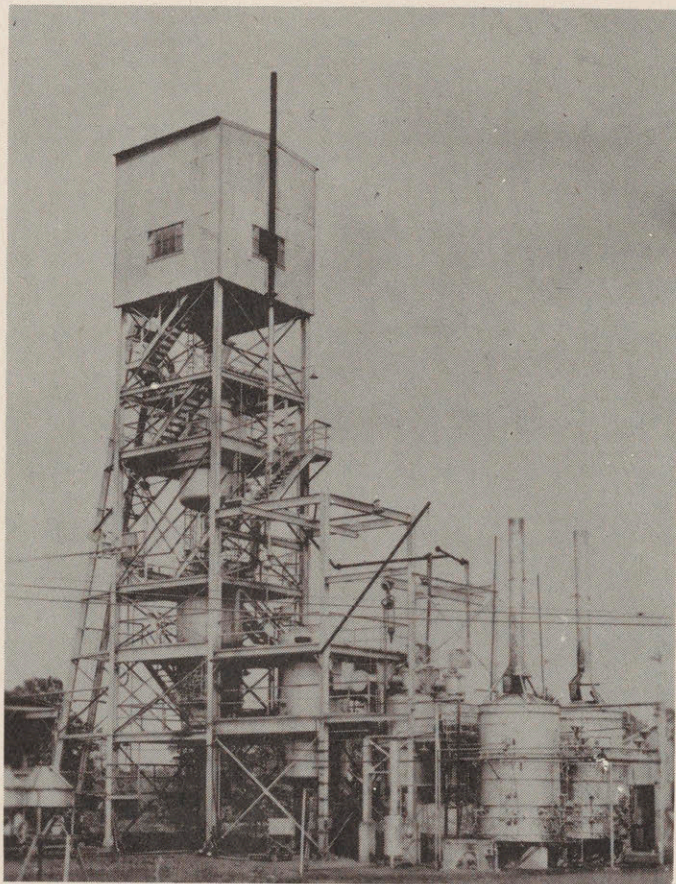


Figure 2. - Sponge-iron plant, Longview, Tex.

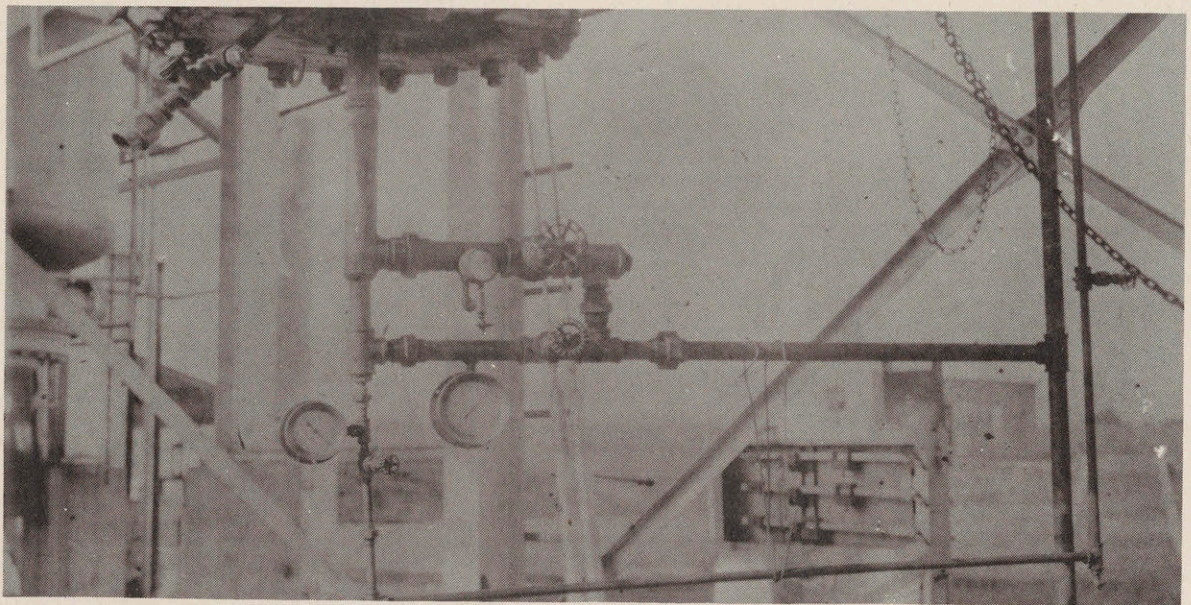


Figure 6. - Surge-tank burner in place.

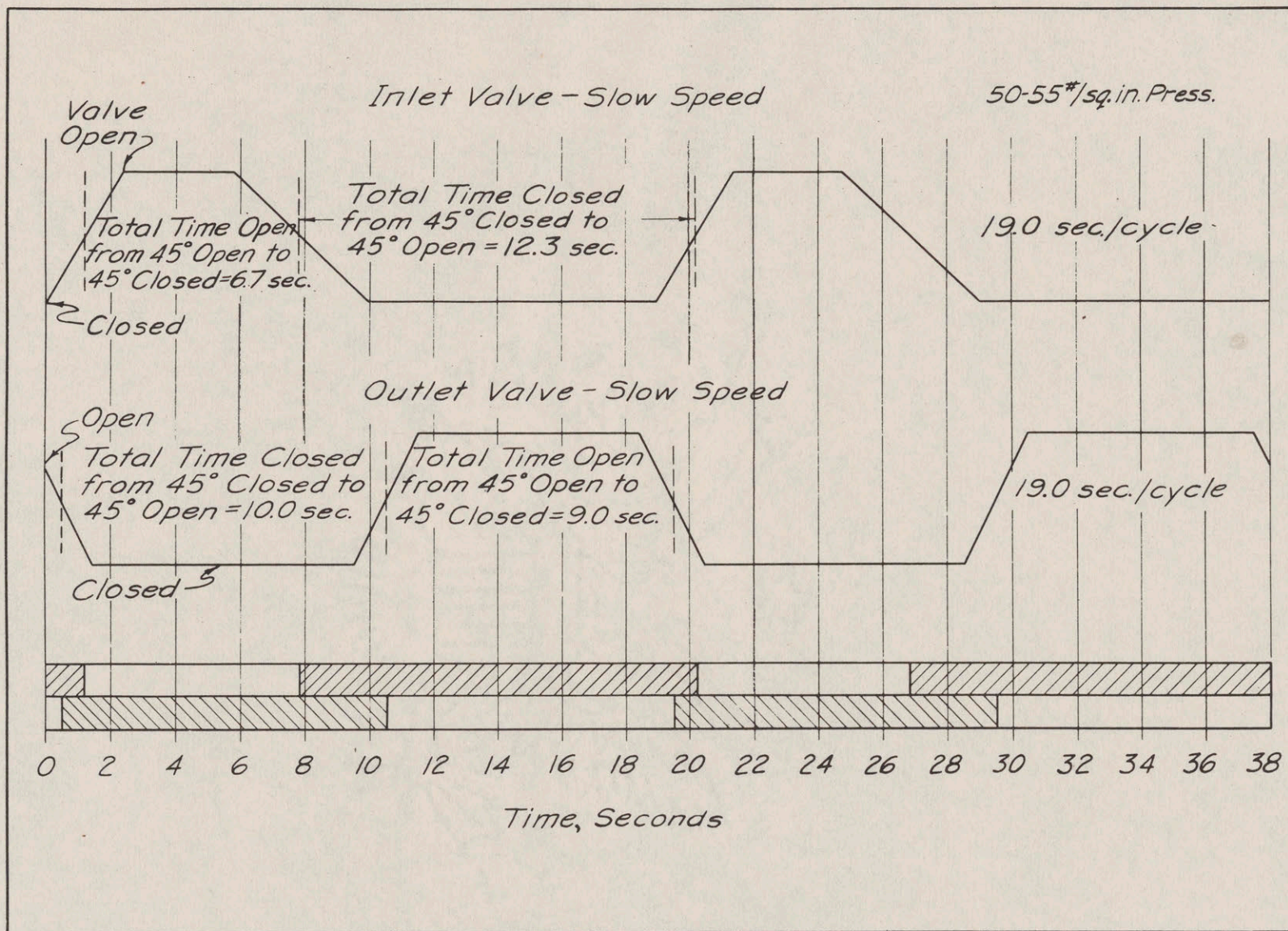


FIGURE 3.- Pulsating-valve cycles.

Reducing gas was prepared by mixing air (preheated to 1,550° F.) with natural gas (preheated to 600° F.) under 40 pounds pressure in a catalytic chamber charged with nickelized alundum cubes. The gas formed by such treatment consists of carbon monoxide, hydrogen, carbon dioxide, oxygen, and nitrogen and small quantities of unchanged methane and heavier hydrocarbons and cracked hydrocarbons.

On leaving the generators, the gas passes into a surge tank, which damps the fluctuation of pressure caused by the pulsating valves and prevents disturbance of the equilibrium of the pressure in the generator. After the reformed gas leaves the surge tank, it passes through the pulsating valve into the reducer and through the ore.

The preheaters and generators are in duplicate. The plant was designed with two natural-gas converter units in parallel, but it was difficult to use both converters at the same time.

In the discussions of operation, temperatures are given in degrees Fahrenheit and pressures in pounds a square inch; endothermic heat reactions are indicated by a minus (-) sign and exothermic reactions by a plus (+) sign.

DESCRIPTION OF PLANT

Natural gas was delivered to the plant at approximately 200 pounds pressure from a gas well several hundred yards distant. The pressure was reduced at the plant to 120 pounds. Two air compressors were used to supply air. The air was heated in a coil of 24 turns, 4 feet in diameter. The tubing used for the coil was type 309 Cb stainless steel, 2-1/4-inch o.d. and 2-inch i.d. Natural gas was heated in 3 turns of a 4-foot-diameter coil of standard 1-1/4-inch pipe. Heat was supplied by gas burners directed on these coils, and the air and gas were heated in their respective coils, enough burners being used to heat the air to 1,155° F. and the natural gas from 550° to 600° F. It was inadvisable to heat the gas to a higher temperature because of possible decomposition of methane, which would cause deposition of carbon in the coil. Because of this relatively low temperature, an alloy coil was not needed for the gas. On the other hand, the coil in which the air was preheated had to be of alloy steel to withstand both temperature and 45 pounds pressure. Both coils were set in a well-insulated cylindrical furnace.

Air and Gas Mixer

The heated air and gas are passed to a mixer, appropriately called a "swirler." A drawing illustrating its position and function is shown in figure 4. Much difficulty was experienced in this phase of the process, and numerous changes were made in the design of the swirler to obtain a more thorough mixture of air and gas. It is very important to have an intimate mixture of air and gas both for effective re-forming of the gas and prevention of damage to the surrounding equipment. The extremely high temperatures in the zone of the swirler necessitated the use of the best heat-resisting alloy obtainable.

The air and gas passed from the swiler into a small mixing chamber, which is in the form of the frustum of an inverted cone. This chamber was constructed of alloy metal rather than refractory, since, when refractory alone was used, it was found that the impingement of the swirling hot gases at high velocity would cause erosion.

Above the mixing chamber was placed a high-temperature refractory plate 6 inches in diameter and 1-1/4 inches in thickness, containing 85 holes 3/8-inch in diameter. It served four purposes: (1) To keep the catalyst out of the mixing chamber, (2) to aid in distributing the gases evenly as they entered the catalytic zone, (3) to increase the velocity of the gases so that the early reaction would not be limited to the very bottom of the catalytic chamber, and (4) to stop the swirling action and cause the gases to enter the re-forming zone in a vertical flow. Attempts to substitute high-temperature alloy screens for the high-temperature refractory plate failed. Very fine, high-grade, alundum cement was satisfactory material for the plate.

Gas Generator and Catalyst

The catalytic chamber was an inverted cone. This shape reduced the velocity of the gas mixture and permitted longer contact with the catalyst. On the other hand, it was necessary that the gases have sufficient velocity to insure that combustion would take place within the catalytic bed and not in the mixing chamber or swirler zone. The chamber itself was constructed of high-temperature fire brick. Details of construction are shown in figure 4.

The catalyst was prepared by soaking 1-inch alundum cubes in a boiling, saturated solution of nickel nitrate. After 4 hours of such treatment, the cubes were removed and heated in an oxidizing flame until all the brown nitrous fumes were expelled. This left the catalyst in the form of nickel oxide on the surface of the alundum carrier and ready for charging into the generator. As much as 1,200 pounds of this catalyst was charged at one time. The weight of the catalyst was 74 pounds a cubic foot, and when in place it contained 50 percent voids.

Gases leaving the re-former passed to the surge tank through a 12-inch pipe lined with 4 inches of insulation.

Surge Tank and Burner

The surge tank resembled a large, compressed-air receiver in a vertical position. It was lined with 9 inches of insulating fire brick and 4-1/2 inches of high-temperature fire brick. The volume was 520 cubic feet. When the pulsating valve opened on the outlet side of the surge tank, the gases were swept into the reducer. When the valve closed, the gases were stored under increasing pressure until the valve reopened.

A burner was installed in the bottom of the surge tank to provide additional heat to the reducing gases by direct combustion into the surge tank. A drawing of the burner is shown in figure 5 and its installation in figure 6 (fol. p. 4). The heat added to the system in this manner helped to bring the

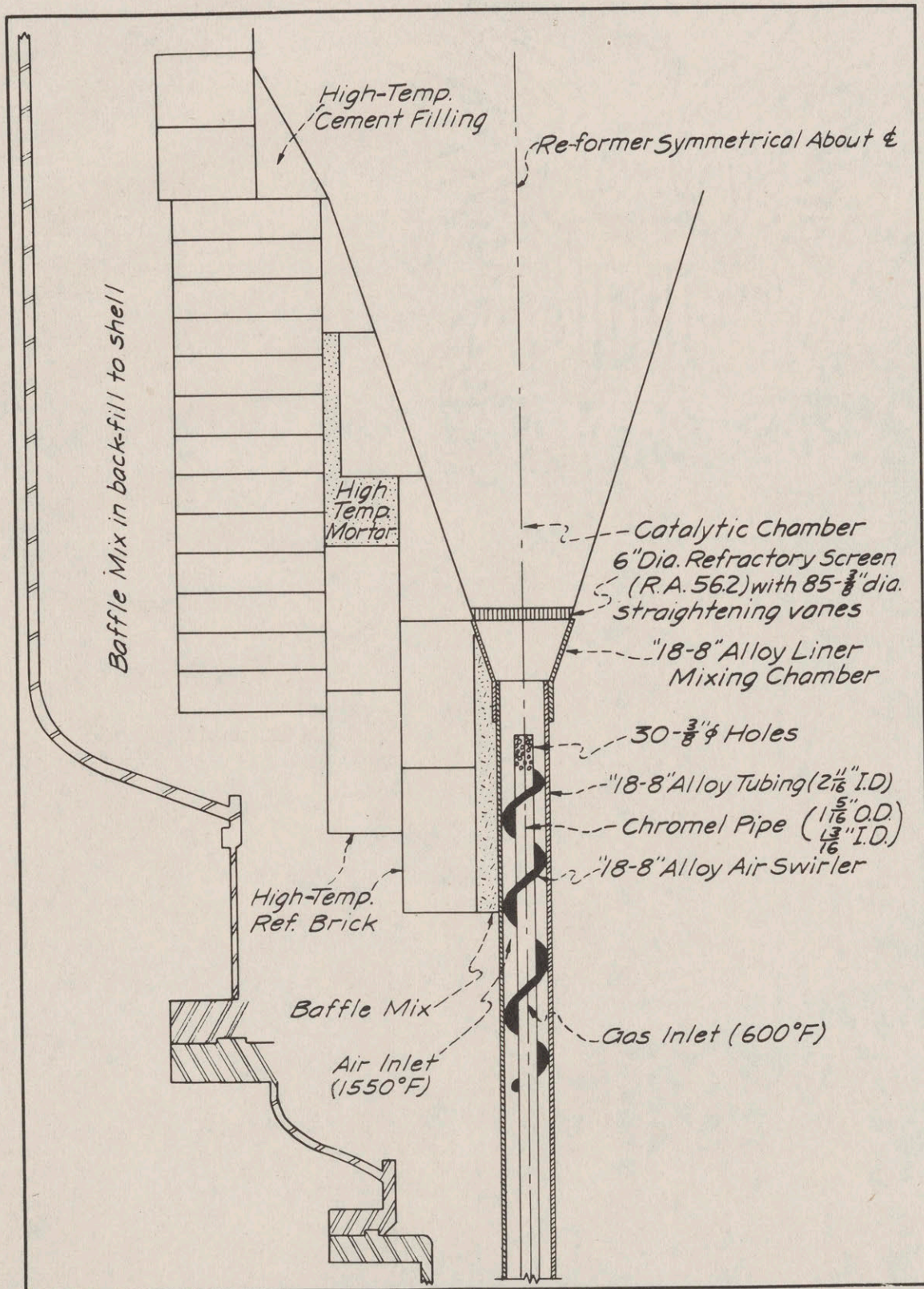
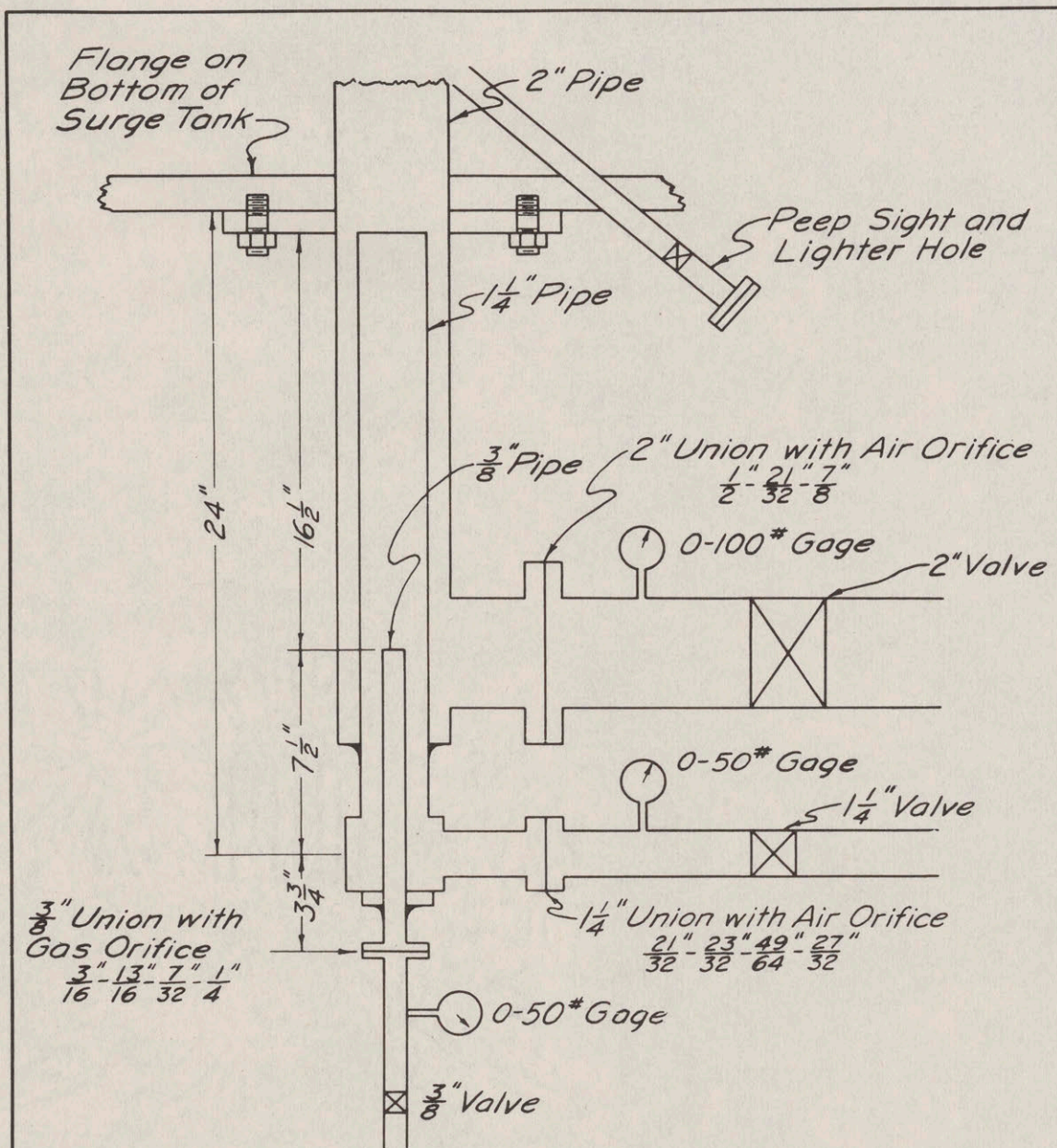


FIGURE 4.- Gas re-former, showing inside construction.



Limit of Orifice Diameter
not to Exceed 0.6 Inside Pipe Diameter

For Gas Orifices of $\frac{3}{16}$ " to $\frac{1}{4}$ " dia.,	use $\frac{3}{8}$ " pipe, I.D. = 0.493
" Combustion Air $\frac{31}{32}$ " to $\frac{27}{32}$ " "	" $\frac{1}{4}$ " " " = 1.380
" Excess Air $\frac{1}{2}$ " to $\frac{7}{8}$ " "	" 2" " " = 2.067

FIGURE 5.- Surge-tank burner.

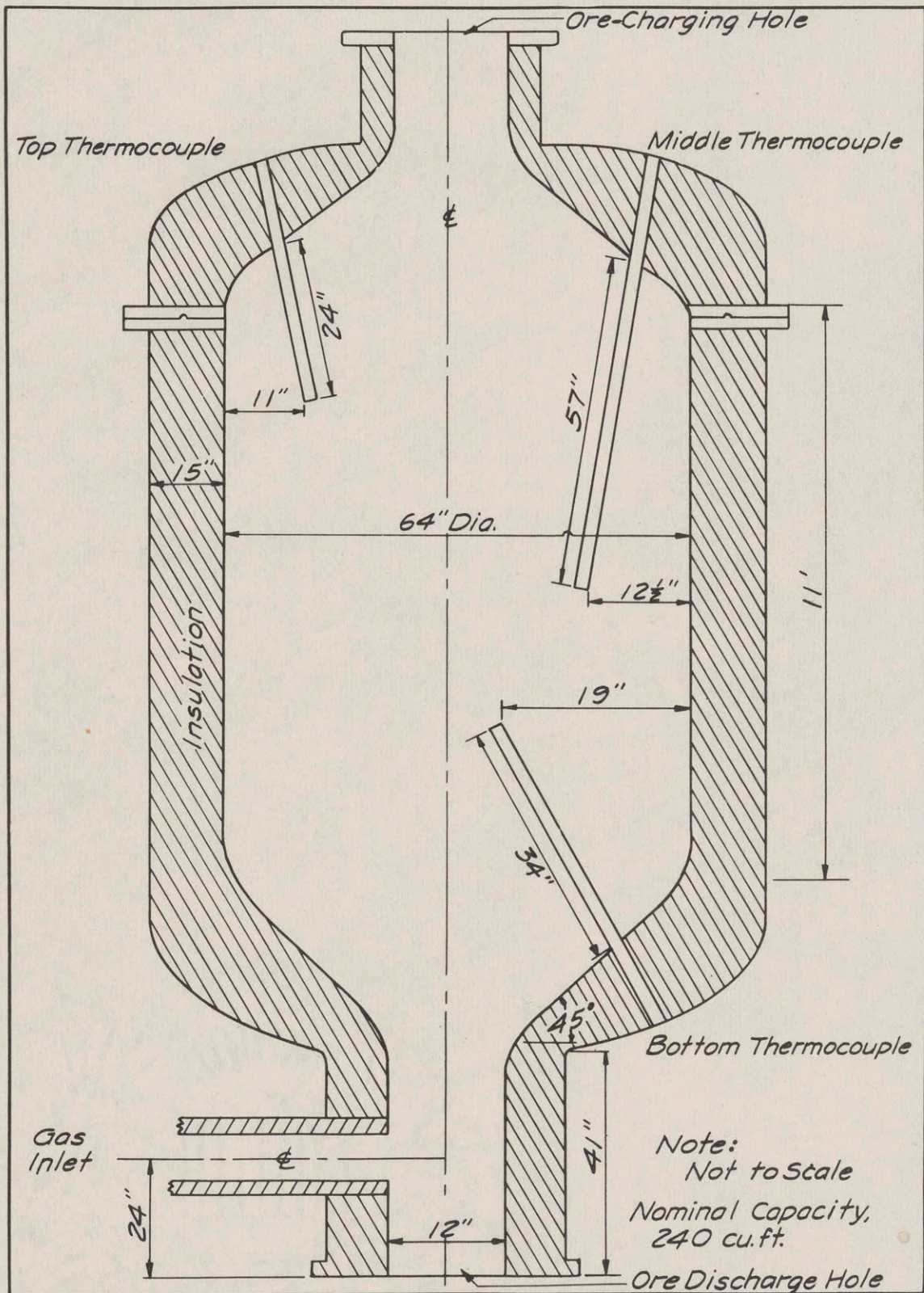


FIGURE 7.- Sketch of ore reduction chamber, showing position of thermocouples.

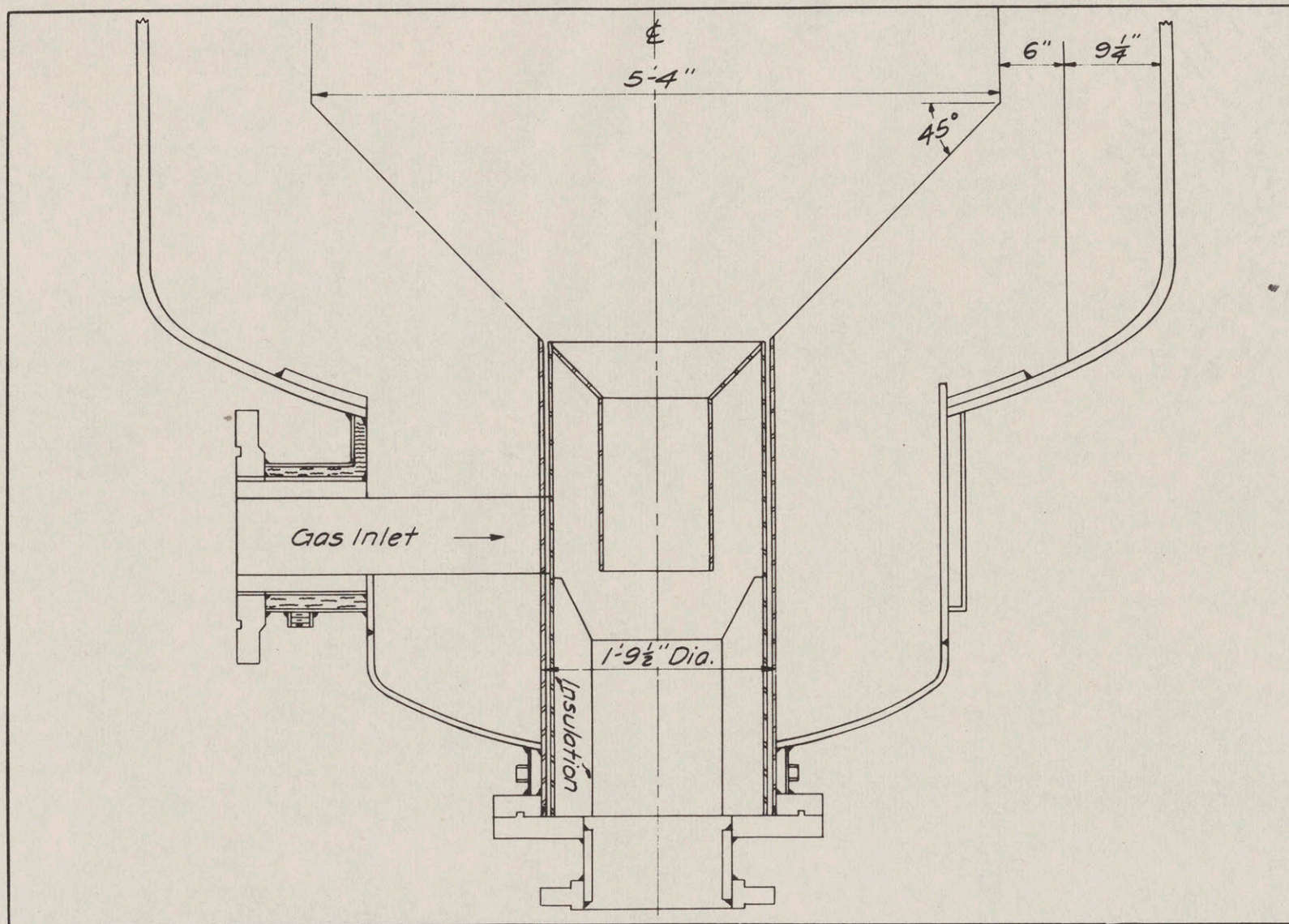


FIGURE 8.- Lower reducer-bottom construction.

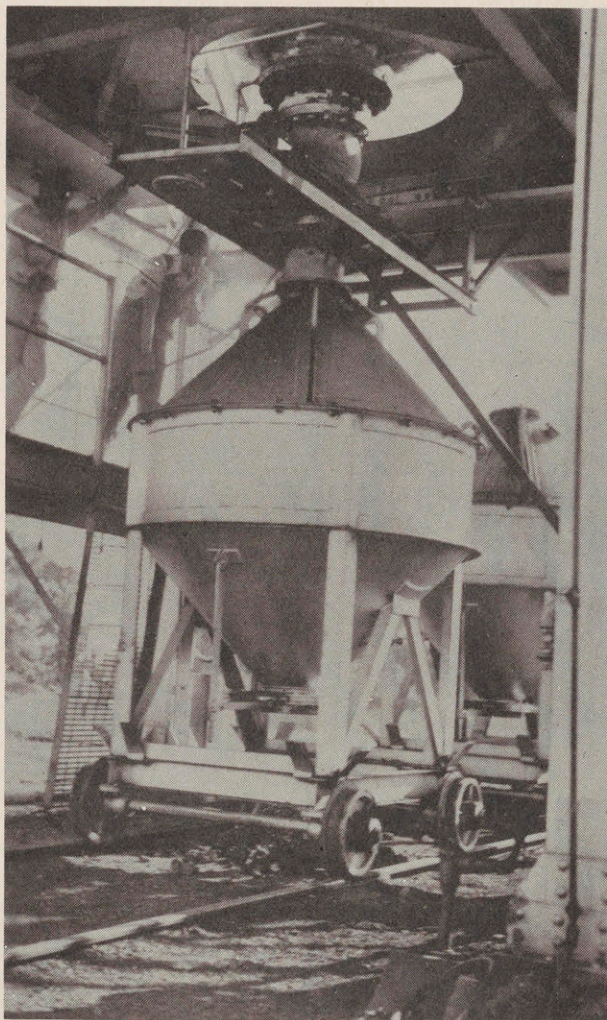


Figure 9. - Sponge-discharging operations.

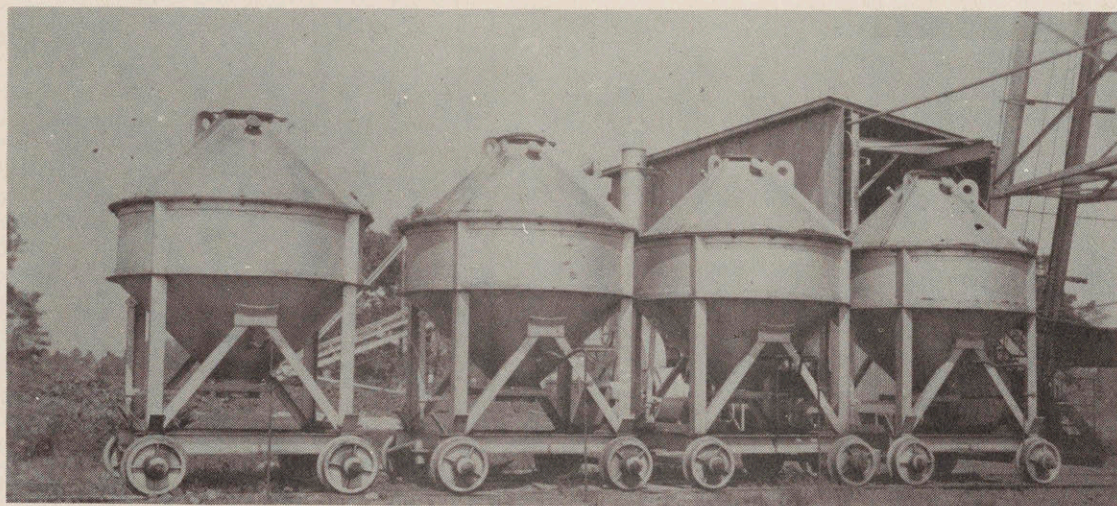


Figure 10. - Sponge-iron storage cars.

ore to reduction temperature, but the addition to the reducing gas of carbon dioxide and water vapor from the products of combustion to the reducing gas was a disadvantage.

ORE REDUCERS

The ore reducer was similar in shape to the surge tank, but its volume was only 240 cubic feet. The cylindrical wall was insulated to a thickness of 15 inches. The bottom was a 45° conical zone cast from insulating material. This slope was made to facilitate the removal of ore at the end of a reduction period. Sketches of the reducer are shown in figures 7 and 8. The plant was originally designed to use two reducers, the second being directly above the first and in series with it. It was intended that the spent reducing gases leaving the lower reducer should pass through the upper reducer, preheating and partly reducing the next charge. By this method of operation, a semicontinuous process might be approached.

The entire plant was covered with aluminum paint for the conservation of heat.

Sponge-Iron Storage Cars

On the completion of reduction the sponge iron was discharged through a 12-inch gate valve in the reducer bottom into three storage cars of 80 cubic feet capacity. A photograph of the sponge-discharging operation is shown in figure 9.

It was necessary to use great care in cooling the sponge iron to prevent reoxidation. While the cars were being filled, natural gas was passed into them to displace the air. (See fig. 10.)

Control Room

Because of the rather large size of the pilot plant and the impracticability of reading instruments in awkward and distant places, most of the instruments were placed on a panel board in a control room where most operating data could be obtained during a reduction period. Gas samples were taken as close to their immediate source as possible. Temperature and pressure readings came from the same point as the gas samples and were relayed to the control room. A gas gravitometer and carbon dioxide continuous recorder also were located in the control room, with provisions for switching the instruments to various points of the system at any time.

The chromel-alumel thermocouples were protected by chromel-type T tubes. Leads from all thermocouples were connected to pyrometers in the control room.

Gas analyses were made by the standard Bureau of Mines method in a Burrell gas-analysis apparatus.

INVESTIGATIONS OF PROCESS

Investigations at Longview were divided into two phases: (1) Gas re-forming and (2) ore reduction.

In previous operations of the Madaras Steel Corp., the gas re-forming equipment was not satisfactory, and the data on its operation were not complete. Attempts to re-form natural gas in a manner similar to that described by Maier^{3/} had not produced a satisfactory gas over a period of time necessary to complete a reduction test. Therefore it was necessary to obtain information regarding the operation of the generators before investigating the reduction of ore.

A series of tests was outlined to determine the most effective operating conditions for the production of reducing gas without too intricate a program. On conclusion of these tests, the gas was not entirely satisfactory.

The requisites of a good reducing gas are:

1. Low carbon dioxide content.
2. Low water-vapor content.
3. High carbon monoxide content.
4. High hydrogen content.
5. Complete or nearly complete decomposition of hydrocarbons.
6. Absence of residual oxygen and illuminants.
7. Temperature of gas, above 1,800° F. but below 2,000° F. If the temperature is too high, the particles of ore agglomerate and discharge is difficult.

The most important factors that influence the process are:

1. Air and gas mixing.
2. Air:gas ratio.
3. Temperatures of preheat.
4. Quantity and velocity of air-gas mixture entering the generator.
5. Pressure in vessel during re-forming.
6. Type, amount, and condition of catalyst.

^{3/} Maier, Chas. G., Sponge-Iron Experiments at Mococo: Bureau of Mines Bull. 396, 1937, 81 pp.

7. Quality of natural gas available.
8. Quality of compressed air available.
9. Design of generator.

Gas Re-Forming

A series of tests was undertaken in which the variables were investigated in the following order:

1. Air and gas rate.
2. Air:gas ratio.
3. Pressure in generator.
4. Air temperature.
5. Depth of catalyst bed.

The least amount of catalyst that would remain undisturbed in the generator was 535 pounds, which was equivalent to the volume of the inverted cone and formed a layer $\frac{3}{4}$ inches in depth.

The following factors were constant in the tests in series 1:

1. Catalyst, 535 pounds.
2. Generator pressure, 45 pounds a square inch.
3. Air:gas ratio, 2.4:1.
4. Air temperature, 1,525° F.
5. Gas temperature, 550° F.

The effect of varying the rate of flow of the air-gas mixture is shown in the following tabulation. The composition of the natural gas used is given on page 19.

Series 1

Variable tested, rate of flow of air-gas mixture

Sample No.	Air flow c.f.m.	Generator top temp., °F.	Gas sp. gr.
17.....	382	1,500	0.671
18.....	369	1,550	.677
19.....	394	1,575	.670
20.....	449	1,600	.680
21.....	498	1,670	.675
22.....	314	1,425	.659
23.....	348	1,520	.672

Gas analysis, percent

	Sample number						
	17	18	19	20	21	22	23
CO ₂	1.2	1.6	2.8	1.8	2.0	0.6(?)	1.6
Illum.5	.1	.5	.3	.4	.4	.3
O ₂7	2.0	.3	.7	.7	3.6(?)	.7
H ₂	32.9	28.6	30.7	29.7	28.8	27.7	31.4
CO.....	15.2	13.6	14.1	14.0	14.3	13.7	15.8
C ₂ H ₆	-	-	-	-	-	-	-
CH ₄	5.6	8.9	10.9	10.5	10.3	8.3	10.1
N ₂	43.9	45.2	40.7	43.0	43.5	45.7	40.1

The results of tests in series 1 indicate that an air rate of approximately 380 c.f.m. yields the best results with the above constants.

In the next series of tests 380 c.f.m. of air was considered a constant and only the air:gas ratio was varied, all other factors remaining same as in series 1.

Series 2

Variable tested, air:gas ratio

Sample No.	Air:gas ratio	Generator top temp., °F.	Gas sp. gr.
24.....	1.98 : 1	1,520	0.680
25.....	2.12 : 1	1,500	.679
26.....	2.19 : 1	1,490	.674
27.....	2.30 : 1	1,485	.665
28.....	2.39 : 1	1,490	.665
29.....	2.48 : 1	1,510	.668
30.....	2.58 : 1	1,510	.671
31.....	2.70 : 1	1,515	.679
32.....	2.82 : 1	1,525	.685

Gas analysis, percent

	Sample number									
	24	25	26	27	28	29	30	31	32	
CO ₂	1.7	1.5	1.8	1.6	1.1	1.3	0.8	1.6	1.8	
Illum.3	.4	.1	.4	.1	.4	-	.2	.2	
O ₂1	.4	1.2(?)	3.7(?)	.4	.3	1.4(?)	.9	1.7(?)	
H ₂	26.1	27.4	30.8	26.2	32.6	33.4	31.3	32.6	28.6	
CO.....	13.5	14.2	13.7	12.7	14.8	15.3	14.1	14.6	13.7	
C ₂ H.....	-	-	2.1	1.8	1.7	3.6	-	.6	2.6	
CH ₄	18.0	16.2	5.7	6.0	6.8	5.3	8.3	5.0	1.8	
N ₂	40.3	39.9	44.6	47.6	42.5	40.4	44.1	44.7	49.6	

The best reducing gas was found in sample 29, which shows that under these conditions the most satisfactory gas is obtained when the air:gas ratio is approximately 2.5 : 1.

The third point investigated was the effect of variation in generator pressure, using as constants the previously determined conditions namely,

1. Catalyst, 535 pounds.
2. Air:gas ratio, 2.5 : 1.
3. Air flow, 380 c.f.m.
4. Air temperature, 1,525° F.
5. Gas temperature, 550° F.

Results are as follows:

Series 3

Variable tested, generator pressure

Sample No.	Generator top pressure lb./sq. in.	Generator top temp., °F.	Gas, sp. gr.
33.....	20	1,485	0.693
34.....	30	1,515	.672
35.....	35	1,540	.672
36.....	40	1,530	.668
37.....	45	1,510	.669
38.....	50	1,515	.670
39.....	60	1,505	.668

Gas analysis, percent

	Sample number						
	33	34	35	36	37	38	39
CO ₂	2.2	1.5	1.6	1.6	1.8	1.5	1.8
Illum.5	.3	.4	.1	.1	.1	.3
O ₂6	-	.6	.7	.1	.5	.4
H ₂	31.4	31.8	33.8	33.6	31.2	33.4	34.2
CO.....	14.0	14.5	14.7	15.3	15.4	15.1	15.7
C ₂ H ₆8	1.1	-	-	1.4	.9	.5
CH ₄	8.4	7.9	7.5	8.1	7.5	6.6	7.7
N ₂	42.1	42.9	44.7	41.6	42.5	41.9	39.4

The data of series 3 indicate that the best gas is obtained at a pressure of 60 pounds a square inch. During operation at this pressure the generator shell became excessively heated in spots, indicating that the gases were penetrating the lining and coming in contact with the shell. Since the quality of the gases produced at 40, 45, 50, and 60 pounds a square inch pressure was nearly constant, 45 pounds was chosen for future tests.

Constants for the fourth series of tests were as follows:

1. Catalyst, 535 pounds.
2. Air flow, 380 c.f.m.
3. Air:gas ratio, 2.5 : 1.
4. Generator pressure, 45 pounds a square inch.

The variable to be tested was the temperature of the air-gas mixture. Since the air and gas preheater coils are in the same furnace, any increase in heat input to raise the air temperature will simultaneously increase the gas temperature.

Series 4

Variable tested, air and gas temperature

Sample No.	Preheat temperature, °F.		Generator top temp., °F.	Gas, sp. gr.
	Air	Gas		
40.....	1,350	535	1,435	0.688
41.....	1,400	575	1,455	.685
42.....	1,450	600	1,460	.678
43.....	1,500	615	1,465(?)	.670
44.....	1,550	625	1,460(?)	.671

Gas analysis, percent

	Sample number				
	40	41	42	43	44
CO ₂	1.5	1.5	1.8	1.6	1.8
Illum.4	-	.2	.1	.4
O ₂1	.4	.8	.7	2.4(?)
H ₂	30.7	30.4	32.3	33.4	30.4
CO.....	13.4	15.3	14.7	14.7	14.0
C ₂ H ₆	-	1.1	.3	-	-
CH ₄	10.9	6.8	9.9	8.6	9.2
N ₂	43.0	44.5	40.0	40.9	41.8

Increases in air and gas temperatures tend to improve the quality of the gas produced. Despite the contamination by oxygen of sample 44, this gas was believed to be the best, and the temperature of 1,550° F. was chosen as a constant for subsequent tests. Higher temperatures for preheating the air were not tried because of the physical limitations of the alloy heater coil and also because higher temperatures might have caused decomposition of the gas in the heater coil.

A review of the data obtained shows the constants determined to be:

1. Air flow, 380 c.f.m.
2. Air:gas ratio, 2.5 : 1.
3. Generator pressure, 45 pounds a square inch.
4. Air temperature, 1,510° to 1,550° F.
5. Gas temperature, 590° to 620° F.

The results of tests in series 4 showed that preheat temperatures in the ranges given produced practically the same quality of gas.

Constants for the minimum practical depth of catalyst bed having been determined, the object of the tests in series 5 was to determine the effect of increasing the amount of catalyst in the generator.

Series 5

Variable tested, depth of catalyst

Sample No.	Catalyst		Generator top temp., °F.	Gas, sp. gr.
	Weight	Depth, inches		
44.....	535	34	1,460	0.671
45.....	635	36	1,615	.680
46.....	735	38	1,600.	.697

Gas analysis, percent

	Sample number		
	44	45	46
CO ₂	1.8	1.4	1.3
Illum.4	.4	.3
O ₂	2.4(?)	.9	.5
H ₂	30.4	30.9	34.2
CO.....	14.0	14.8	15.1
C ₂ H ₆	-	.1	.1
CH ₄	9.2	8.7	10.5(?)
N ₂	41.8	42.8	38.0(?)

Percentages of methane recorded in tests in series 5 and 6 are questioned because the percentages of hydrogen and carbon monoxide are high. Methane, and to a small extent ethane, is the source of hydrogen and carbon monoxide, and the volume of methane ought to decrease as the volumes of hydrogen and carbon monoxide increase. The percentage of nitrogen is questioned because it is obtained by difference; and, if methane is too high, nitrogen is too low.

It is apparent that an increase in depth of catalytic bed improved the quality of the gas. It seemed possible that a further increase in depth of bed might decrease the temperature of the gas. The quality of the gas produced from a bed 38 inches in depth was the best obtained in the series of tests made up to this point; because of limitation of time, no further increases in depth of catalyst bed were investigated. It did seem advisable to test the constants previously established with the newly determined depth to bed to determine if the same relationships existed.

The first variable tested with the deeper catalyst bed was the air flow. Constants used in the tests were:

1. Catalyst, 735 pounds, 38 inches depth.
2. Air:gas ratio, 2.5 : 1.
3. Generator pressure, 45 pounds a square inch.
4. Air temperature, 1,450° F.
5. Gas temperature, 550° F.

Series 6

Variable tested, air flow

Sample No.	Air flow, cu. ft./min.	Generator top temp., °F.	Gas, sp. gr.
46.....	380	1,600	0.679
47.....	410	1,600	.684
48.....	455	1,655	.681
49.....	493	1,710	.688

Gas analysis, percent

	Sample number			
	46	47	48	49
CO ₂	1.3	1.9	1.8	1.5
Illum.3	.3	-	-
O ₂5	.9	.4	.2
H ₂	34.2	32.7	31.0	30.0
CO	15.1	14.0	14.9	15.3
C ₂ H ₆1	-	.9	-
CH ₄	10.5(?)	9.4	9.4	10.1
N ₂	38.0(?)	40.8	41.6	42.9

The best rate of flow is approximately 380, cubic feet of air a minute.

The effect of varying the air:gas ratio was tested with the following results.

Series 7

Variable tested, air:gas ratio

Sample No.	Air:gas ratio	Generator top temp., °F.	Gas, sp. gr.
50.....	2.22 : 1	1,610	0.681
51.....	2.32 : 1	1,640	.676
52.....	2.41 : 1	1,640	.678
53.....	2.45 : 1	1,640	.671
54.....	2.60 : 1	1,650	.666
55.....	2.70 : 1	1,645	.678
56.....	2.78 : 1	1,645	.690

Gas analysis, percent

	Sample number						
	50	51	52 ^{1/}	53	54	55	56
CO ₂	1.9	1.4		1.5	1.5	1.7	2.1
Illum.3	.3		.2	.1	.3	.3
O ₂7	.7		.5	.8	.6	.7
H ₂	30.3	30.9		32.1	33.0	32.3	31.6
CO	15.0	15.5		16.3	16.5	16.4	15.2
C ₂ H ₆	-	-		-	-	-	-
CH ₄	12.7	11.2		8.6	7.4	7.5	7.1
N ₂	39.1	40.0		40.8	40.7	41.2	43.0

^{1/} Sample contaminated.

Results of tests in series 7 show that there is no appreciable difference in the quality of the gas when the air:gas ratios are between 2.5 : 1 and 2.7 : 1. However, during test 56, with an air:gas ratio of 2.78 : 1, carbon deposited on the catalyst and caused excessive back pressure. For converting the natural gas available in Longview the theoretically correct air:gas ratio is 2.5 : 1. Therefore, it was decided to use this ratio as a constant in future tests.

Because of mechanical conditions it was impossible to maintain an unvarying air:gas ratio throughout a run.

The final variable to be tested was the generator pressure. For this series of tests the following constants were used:

1. Catalyst, 735 pounds, 38 inches.
2. Air flow, 380 c.f.m.
3. Air:gas ratio, 2.5 : 1.
4. Air preheat, 1,540° F.
5. Gas preheat, 610° F.

Series 8

Variable tested, generator top pressure

Sample No.	Generator top pressure lb./sq. in.	Generator top temp., °F.	Gas, sp. gr.
58.....	25	1,600	0.690
59.....	30	1,610	.690
60.....	35	1,610	.685
61.....	40	1,590	.670
62.....	45	1,600	.670

Gas analysis, percent

	Sample number				
	58 ¹ / ₁	59	60	61 ¹ / ₁	62
CO ₂		1.4	1.7		1.6
Illum.4	.2		.1
O ₂6	1.2		.6
H ₂		31.6	31.7		32.7
CO.....		15.8	15.6		15.8
C ₂ H ₆		-	-		-
CH ₄		8.7	7.8		8.5
N ₂		41.5	41.8		40.9

¹/₁ Sample contaminated.

Gas of the best quality was produced at an operating pressure of 45 pounds.

Two short tests were made to determine the effect of increasing the temperature of the gas. Gas temperatures of 650° and 685° F. were tried, but the quality of the gas produced was very inferior.

Some Operating Difficulties

On completion of the series of tests, it was decided to determine whether the gas generator would produce gas of constant quality for a period long enough to complete an ore-reduction test. Up to this time, the generator had not been in continuous operation for more than 16 hours, but calculations indicated that the generator would have to function properly for more than 24 hours if any data were to be obtained on a continuous operating schedule.

When continuous operation was attempted, numerous difficulties were encountered. The first of these was contamination of the natural gas. Ordinarily the gasoline would be present as a vapor and as such presented no difficulties, but in cold weather it condensed and accumulated at various points in the natural-gas line until the liquid was blown in surges into the gas-preheating system and onward to the generator. When this condition arose, the proper air:gas ratio could not be maintained. Oxygen was deficient, and carbon was deposited in the bottom of the generator. This increased the back pressure, as the carbon retarded the flow of the air and gas. Excessive deposition of carbon also poisoned the catalyst and promoted channeling of the gases. If the air:gas ratio was increased the excess air burned the carbon, and fusion of all carbon-coated surfaces would result. A gasoline scrubber to remove the gasoline was designed, and thereafter little trouble could be attributed to this source.

During the period of investigation of continuous gas re-forming, another difficulty was experienced in the bottom of the generator. Immediately above the gas mixing chamber, an 18-8 alloy screen was employed to support the catalyst in the cone of the re-former. This screen could not withstand the temperatures in that area but fused after a few hours of operation and was blown into the catalyst chamber by the intruding air and gas. Fusion of the catalyst was promoted by fluxing action of the melted alloy. This destroyed the catalyst and necessitated its removal before operations could be continued. To solve this problem, a refractory screen was made from a high-grade alundum cement. The cement was mixed with water to the consistency of putty and rammed into a mold 6 inches in diameter and 1-1/4 inches in depth. After this had dried at 212° F. for 24 hours, 85 holes, 3/8 inch in diameter, were drilled through the semidried screen. When the holes had been drilled, the screen was placed in a muffle and gradually heated to 2,000° F., at which temperature it was cured for 48 hours.

The swirler that mixed the air and gas also caused trouble. At the top of the swirler, 30 holes 1/16 inch in diameter were drilled for the outlet of the gas. In numerous tests, it was found that the top of the swirler

and areas within an 8-inch radius fused from extreme temperatures. Although the cause of fusion was not known definitely, it was thought that the gas outlet holes were so small that the gas came out with sufficient velocity to cut entirely across the air stream and impinge on the alloy-tubing liner, resulting in surface combustion that produced enough heat to fuse the refractory. To rectify this condition the gas-outlet holes were enlarged from 1/16-inch to 3/8-inch diameter, thus lowering the gas velocity. No further difficulties were experienced from this source.

When these difficulties had been overcome, the operating characteristics of the generator were slightly different. For the purpose of further decreasing the amount of methane in the re-formed gas, the amount of catalyst in the converter was increased to 1,200 pounds, or a depth of 62 inches. This added amount of catalyst decreased the methane content and improved the reducing gas slightly. Additional amounts of catalyst had no beneficial effect on the quality of the gas.

Continuous operation of the generator was never achieved. Accumulation of carbon on the catalyst gradually destroyed its effectiveness. The use of steam to prevent formation of carbon or to remove that already formed did not prove satisfactory. After periods of operation, the carbon was removed by shutting down the generator and carefully admitting air to burn out the carbon.

The more important data obtained from the investigation of gas re-forming have been placed on graphs for comparison of results and are shown in figures 11, 12, 13, and 14.

Comparison of Theoretical Re-forming and Actual Results

Computations for both theoretical re-forming and for the actual results obtained at Longview are given in the following pages. It is known that the extent of the changes in composition of the gas will depend on several factors, such as the catalyst and the relative concentration of the constituent gases, but any attempt to consider such factors would lead to such great complexity as would detract from interest in the main problem - reduction of iron ore. The discussion of theory shows the methods of computation and the results that might be approximated in practice.

Pressure 41.44*/sq. in.
Air Flow 380 c.f.m.
Catalyst 535 lb.

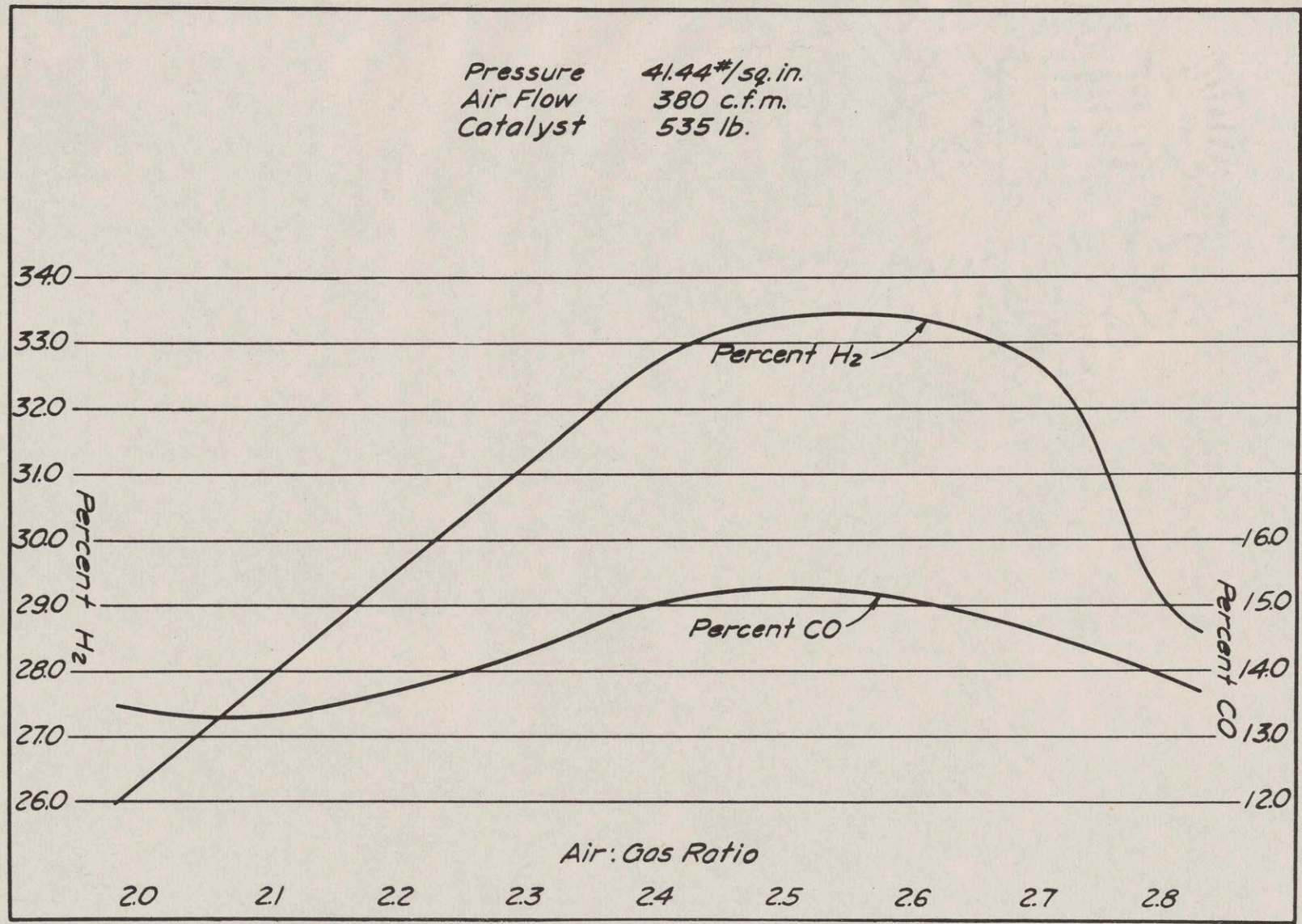


FIGURE 11.- Percent reducing gases vs. air : gas ratio.

FIGURE 12.- Percent reducing gases vs. generator pressure.

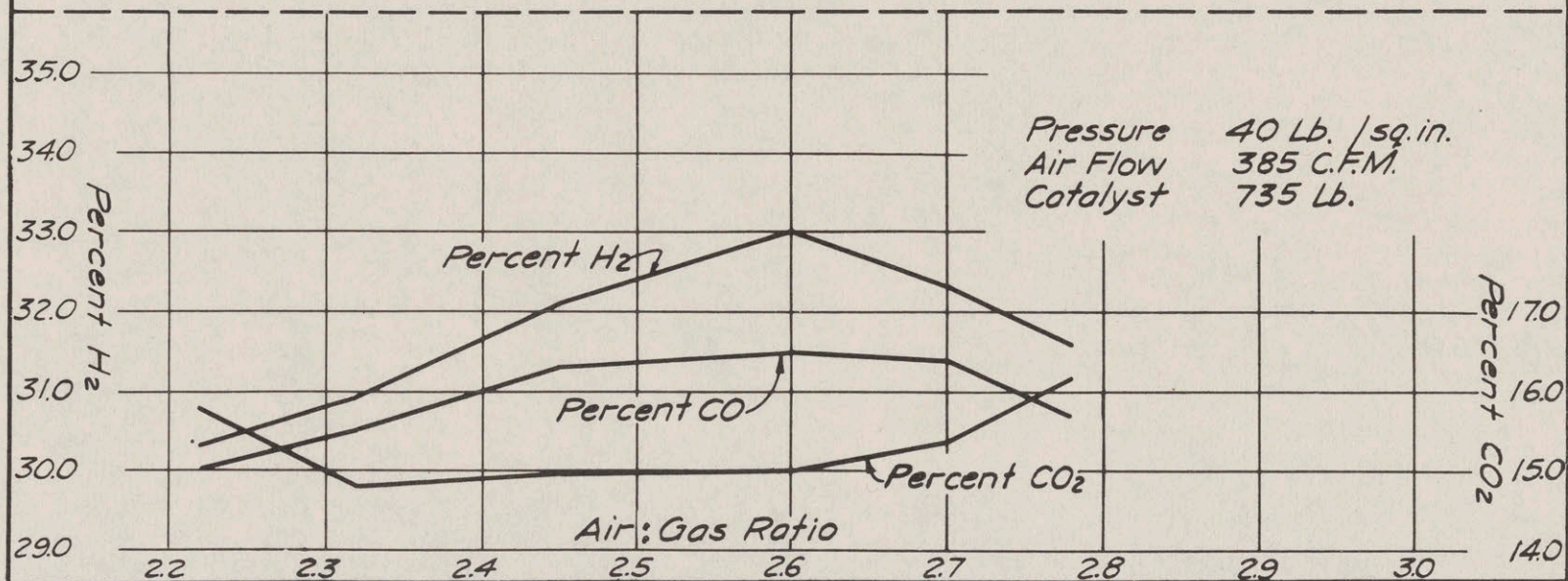
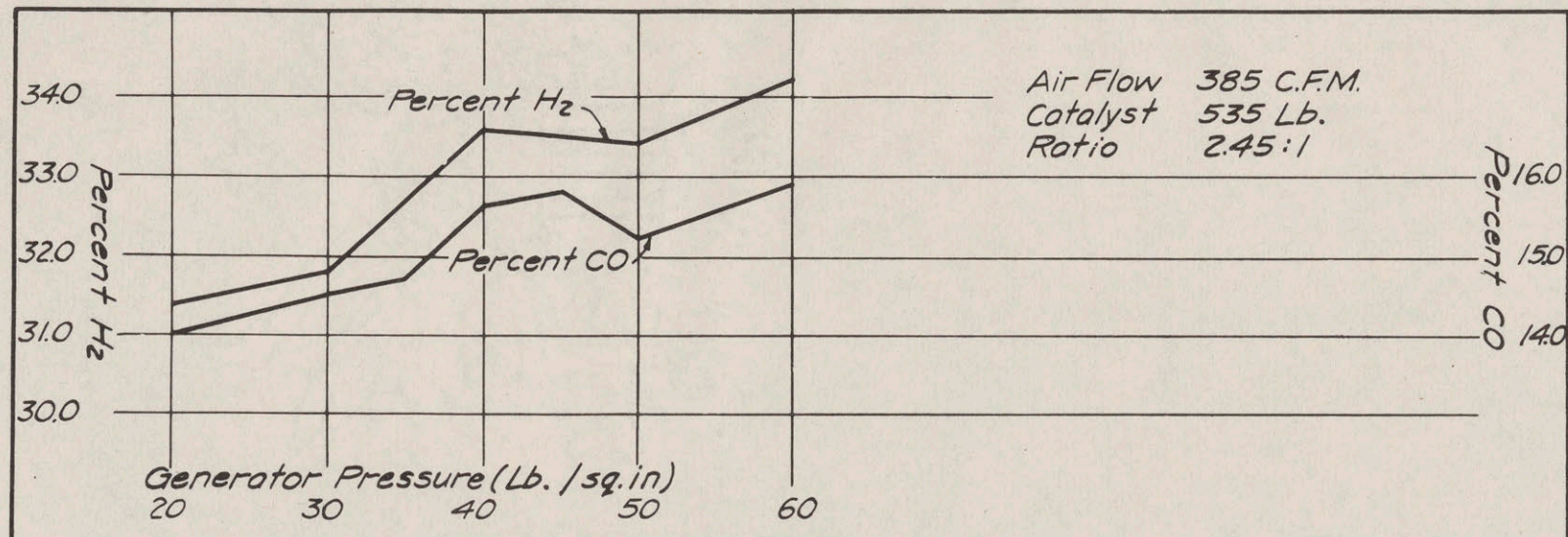


FIGURE 13.- Percent reducing gases vs. air: gas ratio.

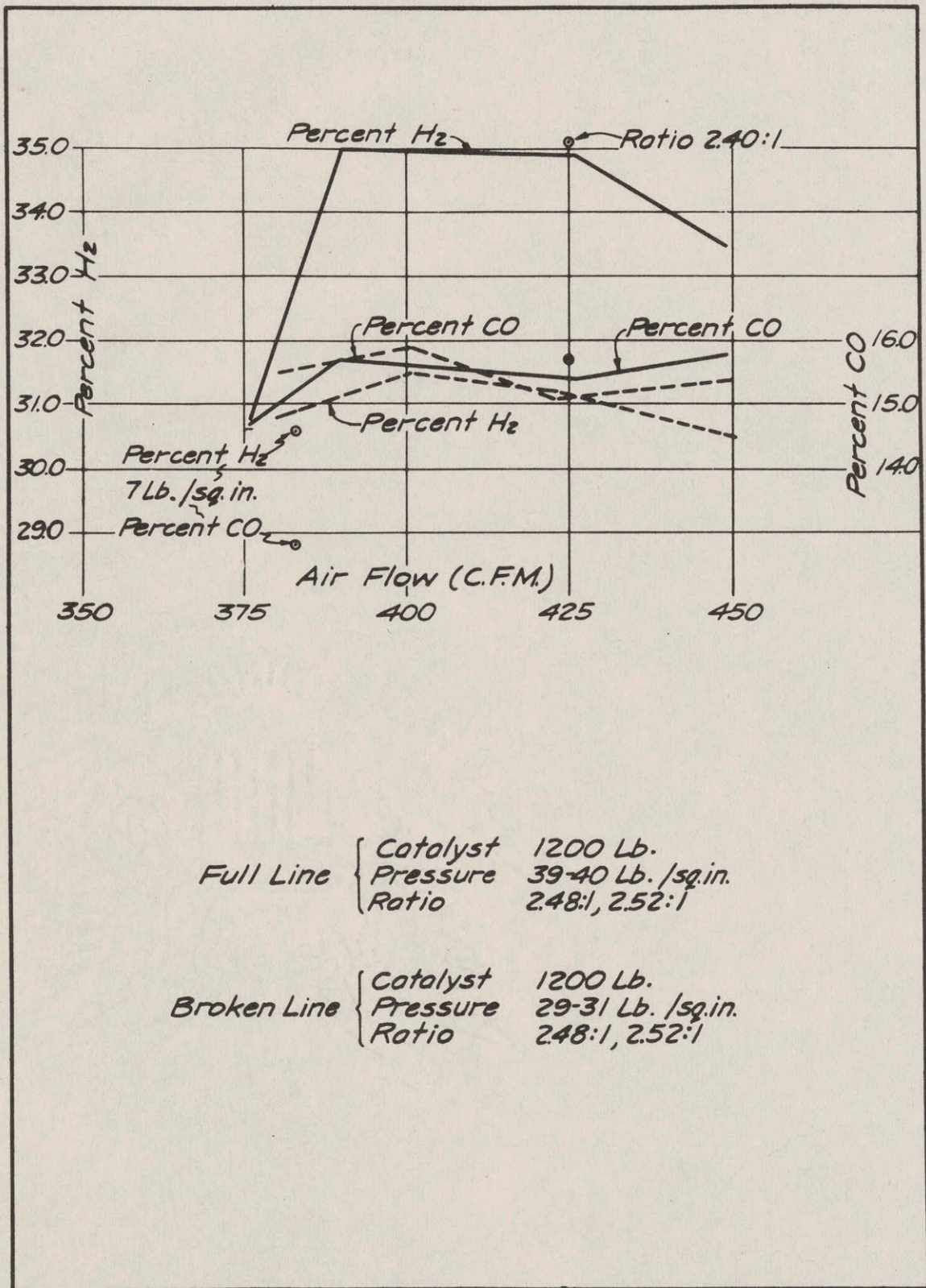


FIGURE 14.- Reducing gas vs. air flow.

Composition of natural gas

	Actual analysis, percent	Analysis used in calculations, percent
CH ₄	92.67	92.67
C ₂ H ₆	4.22)	-
C ₃ H ₈77)	-
C ₄ H ₁₀40) 5.64	5.64
C ₅ H ₁₂19)	-
C ₆ H ₁₄06	-
CO ₂	1.30	1.30
N ₂39	.39
	100.00	100.00

Computation is based on 1 cubic foot of natural gas. All hydrocarbons heavier than methane are considered together as ethane.

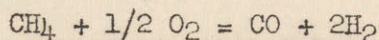
Negative sign indicates endothermic reaction.

Positive sign indicates exothermic reaction.

For simplicity in calculation, it is assumed that carbon is in the gaseous state.

Calculations are on the basis of 1 cubic foot of natural gas and the necessary amount of saturated air to combine with it. The calculations of volumes are as follows:

For methane:



$$\text{Cubic feet: } 0.9267 \quad 0.4633 = 0.9267 \quad 1.8534$$

$$\text{Air required: } \frac{0.4633 \times 100}{21} = 2.2062$$

For ethane:



$$\text{Cubic feet: } 0.0564 \quad 0.0564 \quad 0.1128 \quad 0.1692$$

$$\text{Air required: } \frac{0.0564 \times 100}{21} = 0.2686$$

Total dry air required for combustion to carbon monoxide = 2.2062 + 0.2686 = 2.4748 cu. ft.

Nitrogen added in air = 2.4748 x 0.79 = 1.9551 cu. ft.

The air used was saturated with water vapor. At the assumed temperature of 60° F., the water vapor in 1 cu. ft. of saturated air is 1.73 percent.^{4/}

^{4/} American Gas Association, Combustion: 1932, p. 40.

R.I. 3925

The volume of saturated air required is $\frac{2.4748 \times 100}{98.27} = 2.5184$ cu. ft.

The volume of water vapor added is $2.5184 \times 0.0173 = 0.0436$ cu. ft.

The volumes of the gases produced by combustion with this volume of air will be as calculated above.

The calculated composition, after partial combustion with 2.5184 cu. ft. of air saturated with water vapor, would be

Gas		Cubic feet	Percent
CO	0.9267 + 0.1128	1.0395	20.47
H ₂	1.8534 + 0.1692	2.0226	39.83
CO ₂	0.0130	.0130	.26
N ₂	0.0039 + 1.9551	1.9590	38.58
H ₂ O	0.0436	.0436	.86
		<u>5.0777</u>	<u>100.00</u>

If stoichiometric relations held true, a gas containing not more than 60.3 percent of combined hydrogen and carbon monoxide could be produced. Equations given for re-forming show that there is an increase in volume from the reactions involved.

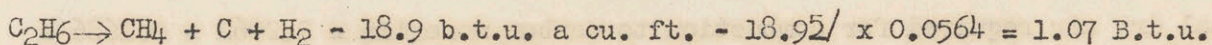
Since the re-forming method was to proceed in a pressure system it was borne in mind that pressure has the greatest effect on that phase of the reaction involving the greatest number of molecules. Hence, pressure would tend to reverse the desired reaction.

On the other hand, increasing the pressure in a gaseous system tends to increase the rate of both the forward and reverse reactions. The presence of a catalyst also increases the rate of reaction and the ideal catalyst will increase the rate of reaction in one direction only.

Taking all these factors into consideration, it seemed that the net effect of pressure would be to produce a re-formed gas at a high rate but not a high conversion.

THERMAL REACTIONS

Decomposition of ethane:



Decomposition of methane:

Total volume of methane is 0.9267 cu. ft. from original gas
plus 0.0564 cu. ft. from decomposition of ethane
0.9831 cu. ft.

5/ Pacific Coast Gas Assoc., Gas Engineer's Handbook: McGraw-Hill Book Co., New York, 1934, p. 354.

$$\text{CH}_4 \rightarrow \text{C} + 2\text{H}_2 - 91.66 / \text{B.t.u. a cu. ft.} - 91.6 \times 0.9831 = 90.05 \text{ B.t.u.}$$

Combustion of carbon to carbon monoxide:

From ethane

$$\begin{aligned} \text{Density of ethane} &= 0.08031 / \text{lb. a cu. ft.} \\ \text{Weight of ethane} &= 0.0803 \times 0.0564 = 0.0045 \text{ lb.} \\ \text{Weight of total carbon} &= 0.0045 \times 0.80 = 0.0036 \text{ lb.} \\ \text{Weight of free carbon} &= 0.0036 \div 2 = 0.0018 \text{ lb.} \end{aligned}$$

From methane

$$\begin{aligned} \text{Density of methane} &= 0.04258 / \text{lb. a cu. ft.} \\ \text{Weight of methane} &= 0.0425 \times 0.9831 = 0.0417 \text{ lb.} \\ \text{Weight of free carbon} &= 0.0417 \times 0.75 = 0.0313 \text{ lb.} \end{aligned}$$

$$\text{Heat of reaction } \text{C} + 1/2 \text{O}_2 = \text{CO}, + 39642 / \text{B.t.u. a lb.}$$

$$3964 \times (0.0018 + 0.0313) = 131.21 \text{ B.t.u.}$$

Net heat of reactions of decomposition of ethane and methane and combustion of free carbon to carbon monoxide:

$$131.21 - 1.07 - 90.05 = 40.09 \text{ B.t.u.}$$

Air leaving the preheater has a temperature of 1,550° F.

Heat losses will cause a temperature drop of not more than 150° F., so that the actual temperature of air entering the generator is about 1,400° F. Natural gas leaving the preheater has a temperature of 600° to 650° F., and it is safe to assume that it reaches the generator at 500° F.

The heat content of air and water vapor above 60° F.¹⁰ would be:

$$\begin{aligned} 2.475 \text{ cu. ft. of air at } 25.09 &= 62.10 \text{ B.t.u.} \\ 0.0436 \text{ cu. ft. of water vapor at } 29.95 &= 1.31 \text{ B.t.u.} \\ \text{Total heat brought in at } 1,400^\circ \text{ F.} &= 63.41 \text{ B.t.u.} \end{aligned}$$

The heat content above 60° F. of the natural gas at 500° F. is:

Gas	Cubic foot	B.t.u. a cu. ft.	B.t.u.
CH ₄	0.9267	13.20	12.23
C ₂ H ₆	.0564	15.37	.87
CO ₂	.0130	10.92	.14
N ₂	.0039	8.12	.03
	1.0000		13.27 B.t.u.

- 6/ Work cited in footnote 5, p. 354.
 7/ Work cited in footnote 5, p. 100.
 8/ Work cited in footnote 5, p. 100.
 9/ Work cited in footnote 5, p. 173.
 10/ Work cited in footnote 5, p. 102.

Net heat of air, water vapor, and natural gas entering the generator is:

$63.41 + 13.27 = 76.68$ B.t.u. for 2.475 cubic feet of air, 0.0436 cubic foot of water vapor, and 1.0 cubic foot of natural gas.

It has already been shown that 40.09 B.t.u. is given off from the re-forming of 1 cubic foot of natural gas. The total heat available to heat the products is $40.09 + 76.68 = 116.78$ B.t.u.

Table 1 which gives the heat content of gases at temperatures within the range of operation, shows that the theoretical calculated temperature is approximately $1,280^{\circ}$ F.

TABLE 1. - Heat in products
(Above 60° F., at 1 atmosphere pressure)

	Cubic feet	B.t.u. at $1,300^{\circ}$ F.		B.t.u. at $1,600^{\circ}$ F.		B.t.u. at $1,400^{\circ}$ F.		B.t.u. at $1,200^{\circ}$ F.	
		1.0 cu. ft.	Total	1.0 cu. ft.	Total	1.0 cu. ft.	Total	1.0 cu. ft.	Total
CO.....	1.0395	32.86	34.16	28.96	30.11	25.09	26.08	21.27	22.11
H ₂	2.0226	33.03	66.81	29.07	58.80	25.16	50.89	21.28	43.04
CO ₂0130	49.46	.64	43.08	.56	36.83	.48	30.74	.40
N ₂	1.9590	32.86	64.37	28.96	56.73	25.09	49.15	21.27	41.67
H ₂ O _l /.....	.0436	36.92	1.61	31.85	1.39	26.95	1.18	22.20	.97
	5.0777		167.59		147.59		127.78		108.19

1/ Sensible heat above 212° F. was used to calculate the heat effect due to H₂O.

To show what actually happened during re-forming, several things must be considered:

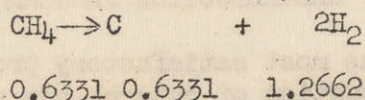
1. Analysis indicated that nearly all of the gases produced contained about 7 percent of unchanged methane. This amounts to approximately 36 percent of the original methane.
2. The temperature of the gases in the re-forming zone of the generator exceeded $2,100^{\circ}$ F.
3. The temperature of gases leaving the generator was always above $1,500^{\circ}$ F. and below $1,650^{\circ}$ F. when equilibrium was reached. Loss of heat by radiation was responsible for the drop in temperature.
4. The pressure in the generator was an important factor in the quality and temperature of the exit gases.
5. The condition and amount of catalyst used in the generator are factors affecting the quality of the gas produced.

6. Although complete re-forming was not obtained, the best gases were made when the required theoretical ratio of approximately 2.5 volumes of air to 1 of gas was used. If either substantially higher or lower ratios were used, the quality of the gases was lowered.

The sum of methane and ethane, considered as methane, in 1 cubic foot of the natural gas is 0.9831 cubic foot. If it is assumed that 0.35 cu. ft. of this does not react, the amount reacting is $0.9831 - 0.35 = 0.6331$ cu. ft. This 0.6331 cu. ft. of methane weighs $0.6331 \times 0.0425 = 0.0269$ pound. The weight of carbon contained in methane reacting is 75 percent of $0.0269 = 0.0202$ pound.

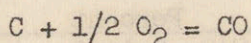
There was added 2.4750 cu. ft. of dry air for each cubic foot of natural gas. This contained $2.4750 \times 0.21 = 0.5198$ cu. ft. of oxygen and $2.4750 \times 0.79 = 1.9552$ cu. ft. of nitrogen.

Since only 0.6331 cu. ft. of methane was decomposed, the reaction

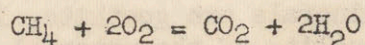


produced only 0.6331 cu. ft. of carbon, considered as gaseous, and 1.2662 cu. ft. hydrogen.

This 0.6331 cu. ft. of carbon required for the reaction



0.3165 cu. ft. of oxygen, and $0.5198 - 0.3165 = 0.2033$ cu. ft. of oxygen remained which could react with methane. Assuming the reaction



the quantities involved would be

$$0.1017 + 0.2033 = 0.1017 = 0.2033.$$

This leaves $0.3500 - 0.1017 = 0.2483$ cu. ft. of unchanged methane.

On the basis of these calculations, the composition of the gas produced would be:

Gas		Cubic feet	Percent
CH ₄	0.2483	0.2483	5.61
CO	.6331	.6331	14.31
CO ₂	.0130 + .1017	.1147	2.59
H ₂	1.2662	1.2662	28.62
N ₂	.0039 + 1.9552	1.9591	44.28
H ₂ O	.2033	.2033	4.59
		<u>4.4247</u>	<u>100.00</u>

This theoretical analysis closely approximates the actual composition of the reducing gas obtained. The percentages of carbon dioxide and water vapor are greater than those actually obtained because the oxygen available was not adequate for complete combustion of undecomposed methane, and some carbon monoxide and hydrogen were produced and less carbon dioxide and water vapor than are shown.

Conclusions on Re-forming of Gas

The re-forming of natural gas under pressure, with the methods used at Longview and with the equipment available, was less satisfactory than had been expected. The inability to produce reducing gas of higher quality was largely responsible for the failure to obtain high reduction of the iron ore in the subsequent tests. Carbon dioxide and water vapor must be eliminated before efficient reduction can be insured. It is obvious that the re-forming of gas under pressure should be investigated more completely.

ORE-REDUCTION PROGRAM

After development of the most satisfactory procedure for production of reducing gas by partial combustion of natural gas and rehabilitation and adjustment of the plant equipment, actual reduction tests were begun. The ore chosen was from the North Basin area, near Linden, Cass County, Tex.

The principal constituents in an average sample of this ore after washing are:

Percent						
Fe	P	S	SiO ₂	Al ₂ O ₃	Free Moisture	Calcination loss
50.1	0.105	0.009	10.08	3.95	1.4	12.9

The principal iron minerals are goethite, limonite, and siderite. Attempts to improve the quality of the material by ore dressing methods in the laboratory of the Bureau of Mines, Rolla, Mo., were not encouraging.

BENEFICIATION TESTS

The most important constituents of the sample tested were as follows:

Analysis, percent							
Fe	Insol.	SiO ₂	Al ₂ O ₃	CaO	P	S	Ig. loss
52.39	11.58	9.61	4.59	0.54	0.128	0.034	11.92

Samples of the ore were crushed and ground to minus 8-, minus 28-, minus 100-, and minus 200-mesh. Each sized portion was roasted for 30 minutes in hydrogen gas at 550° C., and the roasted portions were concentrated in a Davis-tube wet magnetic separator. In these tests the minus 200-mesh material contained some magnetic iron oxide in very fine form, and during operation of the Davis tube this very fine magnetic material was whipped beyond the range of the magnetic field and washed into the

nonmagnetic tailing, resulting in a high-iron tailing and a loss of iron. The best test was on minus 100-mesh material, and the data obtained are as follows:

TABLE 2. - Roasting and magnetic separation test, minus 100-mesh ore

Products	Wt., percent	Analysis, percent								Percent of total Fe
		Fe	Insol.	SiO ₂	Al ₂ O ₃	CaO	P	S	Ig. loss	
Magnetic....	95.85	61.93	8.57	7.81	4.48	0.37	0.147	0.021	-1.11	99.5
Nonmagnetic.	4.15	7.11	80.06	78.06		1.65				0.5
Composite...	100.00	59.66	11.54	10.73		.42				100.0
Roasted										
head, minus 100-mesh..		60.85	11.28	10.45	4.48	.50	.133	.025	-2.14	
Unroasted										
head.....		52.39	11.58	9.61	4.59	.54	.128	.034	11.92	

The test showed that an appreciable amount of the acid-insoluble material in the ore could be removed by roasting and magnetic separation, but that the major part of the insoluble material was too intimately associated with the iron minerals to allow production of a low-insoluble magnetic product, even with fine grinding. The iron-bearing bauxite in the ore evidently became magnetic during the roasting process and was concentrated with the magnetic portion. This probably accounts for the greater part of the alumina in the magnetic portion.

The phosphorus content of the ore was high, and test results show a slight concentration of this element in the magnetic portion. This high phosphorus content may make the ore undesirable in the production of sponge iron, especially if the sponge product is to be melted and refined in an electric furnace.

Beneficiation of this ore with the object of providing a higher grade sponge product was not encouraging. Magnetic tests showed that concentration after reduction would be no more helpful than before reduction.

In the early investigations, the ore was prepared by crushing in a hammer mill to minus 3/4-inch. This product was thoroughly mixed with about 4 percent coke and 8 percent water and sintered. The sinter was roll-crushed and sized to minus 1-inch plus 3/16-inch to prepare the charge for the reducer. The minus 3/16-inch sinter was stock-piled because resintering of the fine material produced a very dense product and often resulted in partial fusion of the ore to a complex iron-aluminum silicate, which was considered unsuitable for efficient reduction.

The sintering process reduced some of the hydrous iron oxide to the magnetic oxide, Fe₃O₄, which, with the loss of volatile matter, increased the iron content of the product to about 60 percent.

A rough sketch of the ore reducer with the positions of the thermocouples is shown in figure 7.

The first three reduction tests were made primarily to determine the operating characteristics of the plant. The loss of heat by radiation presented one of the major problems of the reduction program. Part of the excess piping through which the reducing gases flowed was removed to decrease the heat loss. The second gas generator was built to double the volume of reducing gas delivered to the ore.

Before reduction, the sintered ore in the reducer was heated to reduction temperature by the products of combustion from a natural gas burner placed in the bottom of the surge tank. This burner is shown in figure 1. Excess air was used during the preheating period. It was originally intended that the surge-tank burner should be used only during preheating of the ore and that reduction should be accomplished entirely by the gas from the generator. The loss of heat was so great that it was necessary to continue to use this burner during reduction. Insufficient air for complete combustion was used at this time. This made it possible to increase the temperature of the reducing gas, but this improvement in temperature was accompanied by some decrease in quality, since carbon dioxide and water vapor from the burner were added to the gas.

The data from runs in which significant information was obtained are given in table 3. The gas analyses and the temperatures are averages for each run.

Run 4. This run provided the first data of important value.

The reducer was charged with 13,000 pounds of ore and heated to 1,600° F at the bottom and at the middle and 1,625° F at the top. Since the reducer was only about half full, the top thermocouple did not record the true ore temperature.

After admission of the reducing gas, the temperature of the ore decreased rapidly, and at the end of the test the average temperatures were:

	°F.
Surge-tank outlet	1,504
Bottom zone	1,336
Middle zone	1,161
Top zone	1,312

The test was terminated after 880 minutes, when no further reduction was being accomplished. This was determined by means of a carbon dioxide recorder connected to the spent-gas line.

TABLE 3. - Summary of data on tests for production of sponge iron at Longview, Tex.

	Run 4	Run 6	Run 8	Run 9	Run 12	Run 13	Run 15	Run 16
Charge, pounds.....	13,000	20,500	18,000	18,000	25,000	25,000	25,000	25,000
Size, inches.....	-1 +3/16	-1 +3/16	-1 +3/16	-1 +5/32	-1 +5/32	-1 +5/32	-1 +5/32	-1 +5/32
Time of reduction, minutes.....	880	900	760	1,280	1,500	1,180	975	1,330
Nat. gas to generators, cu. ft.	286,380	282,600	249,280	216,320	270,000	240,405	298,350	206,150
Nat. gas to preheaters, cu. ft.	49,561	34,358	42,170	41,310	63,045	72,033	45,132	41,880
Nat. gas to surge-tank burner, cu. ft. ...	6,688	6,840	11,324	19,072	22,350	17,582	14,528	34,580
Total nat. gas used, cu. ft.	342,629	323,798	302,774	276,702	355,395	330,020	358,010	282,610
Total iron in charge, percent.....	58.2	58.2	54.1	50.1	50.1	50.1	50.1	50.1
Total iron in charge, pounds.....	7,566	11,931	9,738	9,018	12,525	12,525	12,525	12,525
Metallic iron in sponge product, pounds ..	4,585	6,503	6,982	7,169	9,807	10,032	9,857	9,782
Total nat. gas used a ton of met. iron .. produced, cu. ft.	149,456	99,585	86,730	77,194	72,477	65,793	72,641	57,782
Nat. gas to generators a ton of met. iron produced, cu. ft.	124,920	86,913	71,406	60,349	55,063	47,928	60,535	42,149
Air:gas ratio to generators.....	2.48:1	2.51:1	2.50:1	2.50:1	2.49:1	2.50:1	2.50:1	2.52:1
Gas pulsations a minute to reducer.....	6	6	6	6	6	6	6	6
Temperature, °F.:								
Surge-tank outlet.....	1,504	1,479	1,560	1,625	1,447	1,444	1,501	1,690
Bottom zone.....	1,336	1,270	1,387	1,412	1,349	1,360	1,388	1,421
Middle zone.....	1,161	1,250	1,310	1,333	1,247	1,327	1,266	1,354
Top zone.....	1,312	1,327	1,323	1,340	1,337	1,383	1,370	1,463
Reducing gas, percent:								
H ₂ O.....	3.0	3.3	3.7	4.8	3.9	3.8	3.8	1/--
CO ₂	1.5	1.5	1.8	2.0	2.0	1.8	2.3	-
Illum.....	.1	.2	.4	.1	-	.1	.1	-
O ₂7	.4	.6	.6	.6	.6	.6	-
H ₂	30.7	30.1	30.3	28.7	29.7	31.7	28.8	-
CO.....	15.3	14.0	15.4	12.0	14.9	15.0	14.8	-
C ₂ H ₆	-	1.0	.0	-	-	-	-	-
CH ₄	8.9	9.4	7.2	7.0	7.3	4.8	5.8	-
H ₂	39.8	40.1	40.6	44.8	41.6	42.2	43.8	-

TABLE 3. - Summary of data on tests for production of sponge iron at Longview, Tex. (Cont'd.)

	Run 4	Run 6	Run 8	Run 9	Run 12	Run 13	Run 15	Run 16
Sponge product, percent:								
Total iron	67.8	67.0	69.3	73.6	73.6	72.3	76.7	75.3
Metallic iron	41.1	36.5	49.7	58.5	57.6	57.9	60.4	58.8
Reduction	60.6	54.5	71.7	79.5	78.3	80.1	78.7	78.1
Insoluble	17.6	17.4	18.2	18.4	18.1	18.0	16.0	15.8
Phosphorus08	.09						
	Run 17	Run 18	Run 19	Run 20	Run 21	Run 22	Run 25	Run 26
Charge, pounds	25,000	33,500	25,000	25,000	25,000	25,000	25,000	25,000
Size, inches	-1 +5/32	-1/2 +5/32	-1 +5/32	-1 +5/32	-1 +5/32	-1 +5/32	-1 +5/32	-1 +5/32
Time of reduction, minutes	1,330	1,330	1,330	1,330	1,680	1,330	960	960
Nat. gas to generators, cu. ft.	212,800	220,780	210,140	211,470	254,600	210,930	154,460	157,440
Nat. gas to preheaters, cu. ft.	41,074	42,899	40,705	39,378	44,745	39,571	23,922	28,025
Nat. gas to surge-tank burner, cu. ft. ...	37,772	26,600	37,772	29,260	43,680	35,245	34,560	48,768
Total nat. gas used, cu. ft.	291,646	290,279	288,617	280,108	343,025	285,746	212,942	234,233
Total iron in charge, percent	50.1	60.5	50.1	50.1	50.1	50.1	50.1	50.1
Total iron in charge, pounds	12,525	20,267	12,525	12,525	12,525	12,525	12,525	12,525
Metallic iron in sponge product, pounds ..	9,231	13,214	10,220	9,406	10,220	10,358	9,757	10,020
Total nat. gas used a ton of met. iron produced, cu. ft.	63,188	43,935	56,481	59,559	67,128	55,174	43,649	46,753
Nat. gas to generators a ton of met. iron produced, cu. ft.	46,106	33,416	41,123	44,965	49,824	40,728	31,661	31,425
Air:gas ratio to generators	2.51:1	2.49:1	2.51:1	2.49:1	2.51:1	2.50:1	2.50:1	2.50:1
Gas pulsations a minute to reducer	3	3	3	3	3	3	3	3
Temperature, °F.:								
Surge-tank outlet	1,643	1,636	1,711	1,595	1,719	1,800	1,499	1,658
Bottom zone	1,418	1,440	1,492	1,417	1,442	1,437	1,373	1,575
Middle zone	1,339	1,416	1,430	1,429	1,438	1,546	1,378	1,494
Top zone	1,423	1,461	1,362	1,398	1,394	1,485	1,343	1,499

R.I. 3925

TABLE 3. - Summary of data on tests for production of sponge iron at Longview, Tex. (cont'd)

	Run 17	Run 18	Run 19	Run 20	Run 21	Run 22	Run 25	Run 26
Reducing gas, percent:								
H ₂ O.....	1/ -	3.6	4.8	4.7	4.1	4.6	3.5	3.6
CO ₂	-	2.0	2.7	2.4	2.3	2.6	1.9	2.1
Illum.	-	.9	.6	.3	.1	-	.1	-
O ₂	-	.8	.3	.4	.4	.4	.1	.2
H ₂	-	26.2	25.8	28.2	25.6	25.0	28.7	26.5
CO.....	-	14.9	14.6	14.5	14.3	13.9	15.9	15.6
C ₂ H ₆	-	-	-	-	-	-	-	-
CH ₄	-	5.4	5.4	5.2	3.9	2.5	5.5	7.1
N ₂	-	46.2	45.8	44.3	49.3	51.0	44.3	44.9
Sponge product, percent:								
Total iron.....	74.1	72.2	76.1	73.6	71.4	74.4	72.1	73.9
Metallic iron.....	54.6	47.1	62.1	55.3	58.3	61.5	56.2	59.1
Reduction.....	73.7	65.2	81.6	75.1	81.6	82.7	77.9	80.0
Insoluble.....	14.7	12.5	14.0	14.0				
Phosphorus.....								

1/ Samples lost.

As long as the percentage of carbon dioxide in the spent gas was substantially greater than that in the reducing gas, reduction was taking place. The difference between these two percentages decreased as the rate of reduction decreased, and when they were substantially the same the reduction had gone as far as was possible under the existing conditions.

The highest temperature reached in the reducer during the reduction was 1,450° F. in the bottom zone. The pressure at the outlet of the reducer pulsed six times a minute from 4 to 30 pounds a square inch.

Screen and chemical analyses of the reduced ore from test 4 are as follows:

Size	Weight, percent	Reduction, percent
-3/4 +3/8 inch	34.9	49.7
-3/8 inch +5 mesh	21.9	59.4
-5 +10 mesh	20.0	62.7
-10 +20 mesh	7.6	69.4
-20 +65 mesh	10.9	65.5
-65 mesh	4.7	52.3

Run 5. This run was only partly completed when the gas from the generators became so poor that it was necessary to stop operations.

Run 6. This run differed from run 4 mainly in the amount of ore charged. During previous preliminary reduction tests when both gas generators were in operation, the velocity of gas admitted to the reducer with each pulsation was so great that the ore was alternately lifted and dropped in the chamber. This pulverized the ore to some extent, and an appreciable amount of the fine material was blown out through the stack. A larger quantity of ore was placed in the reducer to prevent lifting of the ore.

The reduction was somewhat lower than in previous tests, which fact is explained by the larger quantity of ore charged to the reducer.

The screen and chemical analyses for this test were as follows:

Size	Weight, percent	Reduction, percent
-3/4 +3/8 inch	17.9	35.3
-3/8 inch +5 mesh	54.6	56.1
-5 +10 mesh	14.1	61.1
-10 +20 mesh	5.3	62.4
-20 +30 mesh	2.3	68.6
-30 +60 mesh	2.6	65.8
-60 mesh	3.2	60.6

The results show that reduction of the fine sizes was greater than that of the coarse sizes on sintered ore.

To increase reduction it was decided to crush the raw ore and size on a 3/16-inch screen. The minus 3/16-inch material was sintered and mixed with the minus 3/4-inch plus 3.16-inch size. The sintered fine ore had high porosity and so little strength that it could not support the weight of the charge without some crumbling. It was necessary to charge the reducer carefully in order to prevent plugging of the gas inlet by an accumulation of fine material.

Run 7. Data obtained had no value because the sponge reoxidized and fused before it could be removed from the reducer.

Run 8. Sintered and raw ore were mixed in equal quantities. Although both gas re-formers were used, lifting of the charge was prevented by placing fire brick and large lumps of ore in the inlet area, so that there was little resistance to the flow of the gas in the smaller area.

The capacity of the surge tank burner was increased and a higher pre-heat of the ore was obtained than in preceding runs. The temperature of the ore just before admitting the reducing gases was:

	°F.
Bottom zone	1,850
Middle zone	1,750
Top zone	1,500

The average temperatures during the reduction are shown in table 3.

A portion of the sample from this test was hand-picked to determine the difference in reduction between sintered and unsintered ore. The reduction of the sintered ore was 63.0 percent, and that of the unsintered ore was 75.8 percent. Although the porosity of the sintered ore was higher than that of the unsintered ore, the density of the surface of the former was so great that it retarded the rate of reduction as compared to that of the raw ore. When the unsintered ore is heated to reduction temperature and the natural water and water of crystallization are driven off, the structure becomes porous and cracks are formed, with the result that more surface is exposed to the action of the reducing gases.

Although this test was informative in regard to the type of material that should be charged to the reducer, the reduction was not satisfactory because the temperature had been too low.

Run 9. Again the capacity of the surge-tank burner was increased to obtain higher temperature of the reducing gases at a possible sacrifice in quality.

When run 9 was started, only one gas generator functioned properly, and the reduction period had to be lengthened because of the smaller supply of gas; however, it was not necessary to double this time. In this and in all subsequent runs, material finer than 5/32 inch was removed from the charge.

Comparison of the results of this test with those of previous tests show the beneficial effect of increased temperature on the consumption of reducing gas in relation to the quantity of metallic iron produced.

Figure 15 shows the reaction temperature, gas temperature, and utilization of carbon monoxide during reduction. Normally the percentage of carbon dioxide in the spent gas would diminish until it approached that in the reducing gas. In other words, when complete reduction was achieved there would be no further reaction, and the spent gas would have the same composition as the reducing gas. The difference is attributed to the fact that the temperatures in the reducer were favorable for the decomposition of carbon monoxide, $2\text{CO} \rightarrow \text{C} + \text{CO}_2$.

The occurrence of this decomposition can be fairly well substantiated by analysis of the sponge product, which often contained as much as 1.5 percent carbon. Another source of carbon is the decomposition of methane according to the reaction, $\text{CH}_4 \rightarrow \text{C} + 2\text{H}_2$. Both of the above reactions are catalyzed by the pressure of iron, especially finely divided iron.

Screen and chemical analyses of test 9, percent

(Sponge-iron-ore reduction)

Size	Weight	Metallic iron	Total iron	Reduction
-1 +3/8 inch.....	16.1	59.6	74.3	80.2
-3/8 +5 mesh.....	48.1	60.8	75.4	80.6
-5 +10 mesh.....	20.0	58.3	73.9	78.9
-10 +20 mesh.....	5.6	59.2	75.8	78.1
-20 +30 mesh.....	2.6	53.2	71.7	74.2
-30 +60 mesh.....	3.8	47.5	65.8	72.2
-60 mesh.....	3.8	39.1	54.2	72.1

In this test the best reduction was in the coarser sizes.

Runs 10 and 11. Data from these runs were discarded because of unsatisfactory operation of the generators and plugging at the reducer inlet. Moreover, the ore in the reducer lifted with each pulsation after about 3 hours reduction time.

It was then decided that at least 25,000 pounds of ore should be used in all future tests to eliminate lifting of the charge. Heat requirements would be greater for the larger charge, but it was considered probable that better utilization of the reducing gas might be achieved if the necessary heat could be supplied. The results of later tests confirmed this belief.

Run 12. One generator was used for the first 22 hours and two generators during the last 3 hours in an effort to increase the rate of reduction at the end of the test, but no improvement resulted.

The duration of the test was increased because of the larger charge and with the expectation of effecting higher reduction. Higher reduction

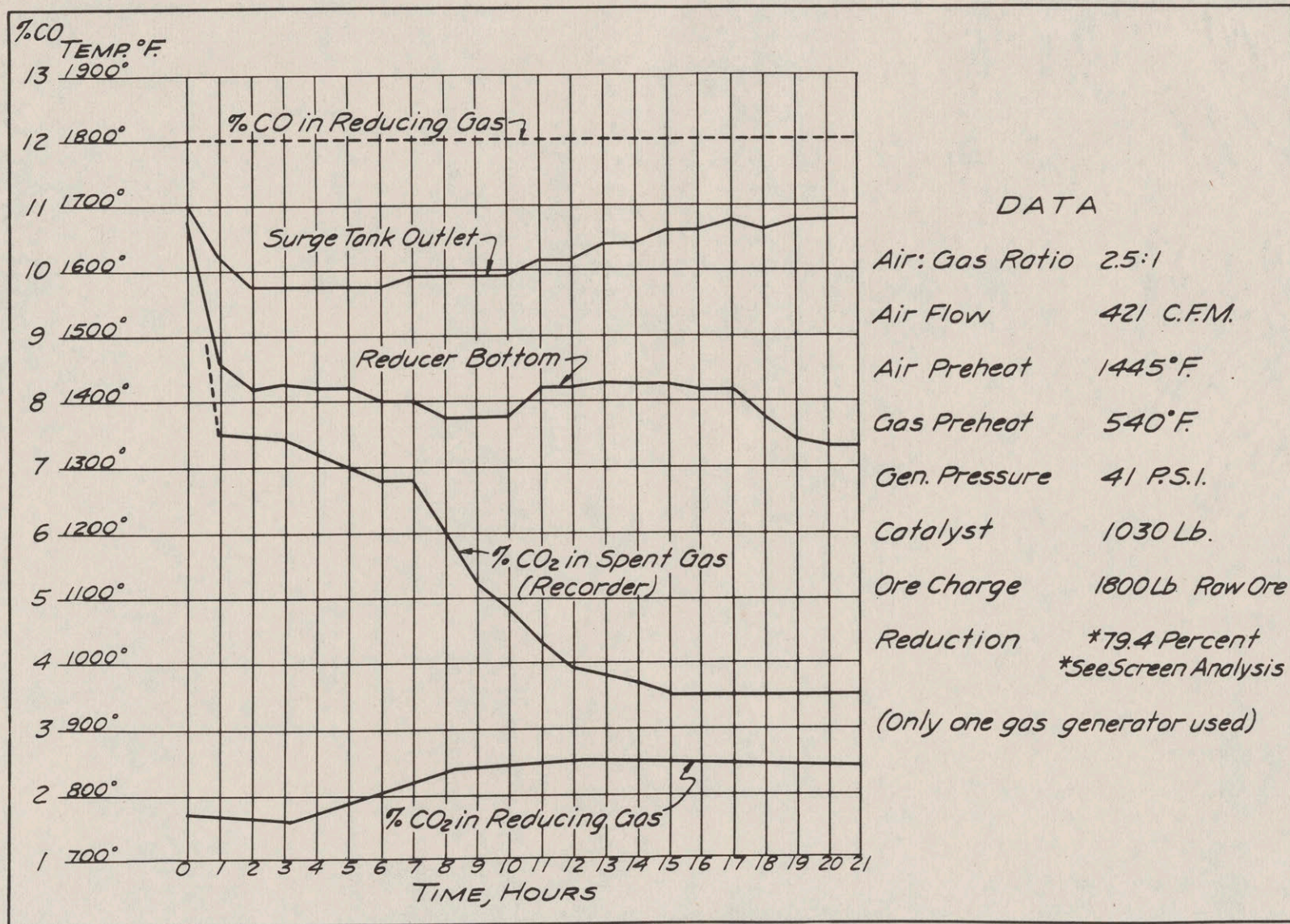


FIGURE 15.- Reduction reactions, ninth test.

was not attained, but the ore was uniformly reduced. In view of the quality of the gas and the temperature, the duration of the test probably was longer than was necessary.

Run 13. The object of run 13, as of run 12, was to determine if the rate of reduction could be increased by operation of both gas generators during part of the run, thus reducing the total time required. Both gas generators were used during the first 6 hours and only one during the remaining 13.6 hours. The percentage reduction obtained was slightly higher, but the gas was of slightly better quality and the temperature in the reducer was a few degrees higher. Toward the end of the first 6 hours, the pressure at the reducer inlet increased, apparently because some fine material had accumulated at the gas inlet and had cemented together.

Removal of the sponge at the conclusion of the test was very difficult because pieces of the sponge had stuck together and to the walls.

Prevention of sticking of the sponge in the reducer was difficult. Placing several cubic feet of broken fire brick mixed with lumps of ore about 4 inches in diameter in the reducer bottom and gas-inlet area only partly solved the problem.

Run 14. Dense sintered ore was placed in the bottom of the reducer, which is the hottest part of the chamber. No data of value were obtained.

Two of the important facts that had been established by operations up to this point were: (1) The reducer should be charged full of ore to prevent alternate lifting and dropping of the charge by the pulsating blast, which caused disintegration of the ore and resulted in much of the powdered material being blown through the upper reducer and out through the stack. (2) When a full charge of 25,000 pounds of raw ore was used, it was almost impossible to use all of the reducing gas from both generators during an entire test, even though the minus 5/32-inch material was screened out. This was due to plugging of the gas inlet in the reducer bottom, which was caused in part by the high velocity of the blast of hot gases in the bottom of the reducer, which tended to pulverize the spongy ore, and in part by compaction of the weak sponge by the burden of the overlying ore.

Run 15. It was thought that the two generators could be used if pulsation were eliminated by throwing the pulsating valves out of operation, since the effect of the blast would be removed. Run 15 was made to determine this.

The pressure in the reducer inlet when two generators were used was 30 pounds a square inch, and the outlet pressure was 4 pounds. The resistance of the charge was 26 pounds. Unfortunately the charge stuck in the reducer and had to be taken out by hand after it was sufficiently cool. A few hand-picked samples from the bottom of the reducer showed 95.7-percent reduction. These specimens were recognized as reduced goethite. An average of the whole charge showed only 78.7-percent reduction. It is reasonable to assume that reduction at the gas inlet would be greatest because this is the zone of highest temperature.

No evidence of channeling was observed, and gas had apparently permeated the charge uniformly.

Temperatures were much too low for rapid reduction. In an attempt to increase the temperature further, alterations were made to allow the burning of more gas in the surge tank at further sacrifice of the quality of the reducing gas.

The results of this test indicate that the value of pulsations of the reducing gas is doubtful. Additional investigation is desirable.

Run 16. Only one generator was used. The top pulsating valve was kept open only wide enough to maintain the pressure at 38 pounds a square inch. The inlet gas pressure was maintained at 48 pounds, giving a 10-pound differential through the ore bed.

Unfortunately all gas samples from this test were lost, but it may be assumed that the average composition of the reducing gas was quite similar to that of run 17.

In this test the rate of consumption of gas in the surge-tank burner was approximately 175 percent of that in the preceding test. The effect of this additional heat is shown by the increase of temperature of the reducing gas from 1,368° F. at the entrance to the surge tank to 1,690° F. at the exit.

Although the reduction time was longer than in all but one of the preceding tests, the volume of reducing gas used was less than in any other.

The percentage of reduction in tests 15 and 16 was practically identical.

Again no evidence of pronounced channeling of the reducing gas was found in the ore bed.

Run 17. For comparative purposes, run 17 was made with conditions as nearly as possible the same as those of run 16, except that the rate of pulsation was decreased from six pulsations a minute, which had been used in earlier tests, to three pulsations a minute because it was thought that this might result in greater utilization of the reducing gases. The reduction obtained in run 17 was only 73.7 percent. Although two tests do not justify a definite conclusion, the results indicate little difference in reductions due to pulsating gas streams.

Run 18. This run was made to determine the efficiency of the utilization of gas on a closely sized calcined ore. An ore that had been given a semimagnetic roast and sized to minus 1/2-inch plus 5/32-inch was tried. The results are shown in table 3.

Although a somewhat lower reduction was obtained in run 18, less natural gas was used to a ton of metallic iron produced. This indicates more efficient utilization of gas. The over-all time was the same as in the previous

run. Average temperatures were a few degrees higher, but hardly sufficient to explain the increased efficiency. More time and gas may have been needed for greater reduction, since more ore was charged because calcined ore weighed more a cubic foot than the raw ore.

It may not be safe to draw definite conclusions from run 18 since two conditions were changed. The ore was closely sized and had been given a semimagnetic roast. The roast had resulted in partial conversion of Fe_2O_3 to Fe_3O_4 , with consequent decrease of the volume of gas necessary for conversion of the oxide to metallic iron.

Runs 19 and 20. These runs were designed to show directly the effect of temperature by comparing a high-temperature, low-quality gas with a lower-temperature, better-quality gas, all other conditions being held as nearly the same as possible. The gas produced in the generators, however, was not of exactly equal quality in the two tests.

Analysis of reducing gases during tests 19 and 20, percent

Gas	Run 19		Run 20	
	Generator	Surge-tank out	Generator	Surge-tank out
H_2O	2.1	4.8	2.1	4.7
CO_2	1.1	2.7	1.1	2.4
Illum.1	.6	.3	.3
O_25	.3	1.0	.4
H_2	33.7	25.8	32.1	28.2
CO	17.3	14.6	16.4	14.5
C_2H_6	-	-	-	-
CH_4	5.9	5.4	4.4	5.2
N_2	39.3	45.8	42.6	44.3
Temperature, 1,231° F.	1,711° F.	1,249° F.	1,595° F.	
Gas burned in surge tank a minute	28.4 cu. ft.		22.0 cu. ft.	

Data from these tests show the effect of rate of the burning of gas in the surge tank burner on the quality and temperature of the gas sent to the reducer. The higher temperature gave greater reduction although the quality of the gas was slightly poorer.

Run 21. To determine the effect of increased reduction time on the extent of reduction, run 21 was continued through 28 hours, other conditions being held constant:

Comparison of results of runs 19 and 21 shows that the extent of reduction was the same in both and that the longer duration of run 21 was without effect. Apparently an equilibrium for the existing temperature and quality of gas had been reached at a reduction of approximately 82 percent.

To determine the efficacy of the process in the treatment of ore in larger sizes, approximately 1,500 pounds of lump ore sized from minus 7 to plus 3 inches was placed on top of the charge for run 21. The reduction was more than 80 percent in all lumps. The photograph in figure 16 shows that a lump of ore was practically the same shape after it had been reduced. The cracks and checks that developed during reduction permitted easy access of the reducing gas to all parts of the lump, and it can be considered that the reduction was practically the same for the lump ore as for the finer sizes.

The treatment of this ore in larger sizes would have the following advantages: Part of the crushing could be eliminated, and the quantity of fine ore to be pelletized or sintered would be smaller; less fine material would be lost through the stack; the resistance of the charge would be reduced, and it might be possible to operate both generators and both reducers, thus increasing the capacity of the plant. The length of time allotted to the program unfortunately did not permit a thorough investigation of the treatment of lump ore.

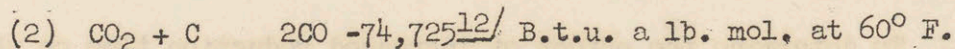
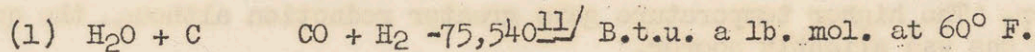
Run 22. This run was made to determine the minimum time required to obtain 80- to 83-percent reduction. This was accomplished in 22 hours 10 minutes.

The generator operated at a slightly higher temperature during this run, and consequently more heat was available throughout the whole system.

During the last 4 hours of this run, a bad leak in an expansion coupling connecting the surge tank with the reducer caused the loss of approximately 25 percent of the reducing gas. Since 82.7-percent reduction was obtained, it was safely assumed that the ore was at that time already reduced to its ultimate state.

Runs 23 and 24. These runs failed because of mechanical difficulties.

A program was then outlined to test the effect of passing the reducing gases through a bed of incandescent coke to convert the water vapor and carbon dioxide to hydrogen and carbon monoxide according to the following water gas and carbon dioxide reduction reactions.



Both of these reactions are endothermic and a proportional amount of heat must be added to sustain the reaction.

The design of the surge tank was such that the inlet reducing gases had to be mixed with the combustion gases and then passed through a bed

^{11/} Work cited in footnote 5, p. 174.

^{12/} Work cited in footnote 5, p. 102.



Figure 16. - Large lump of reduced ore.

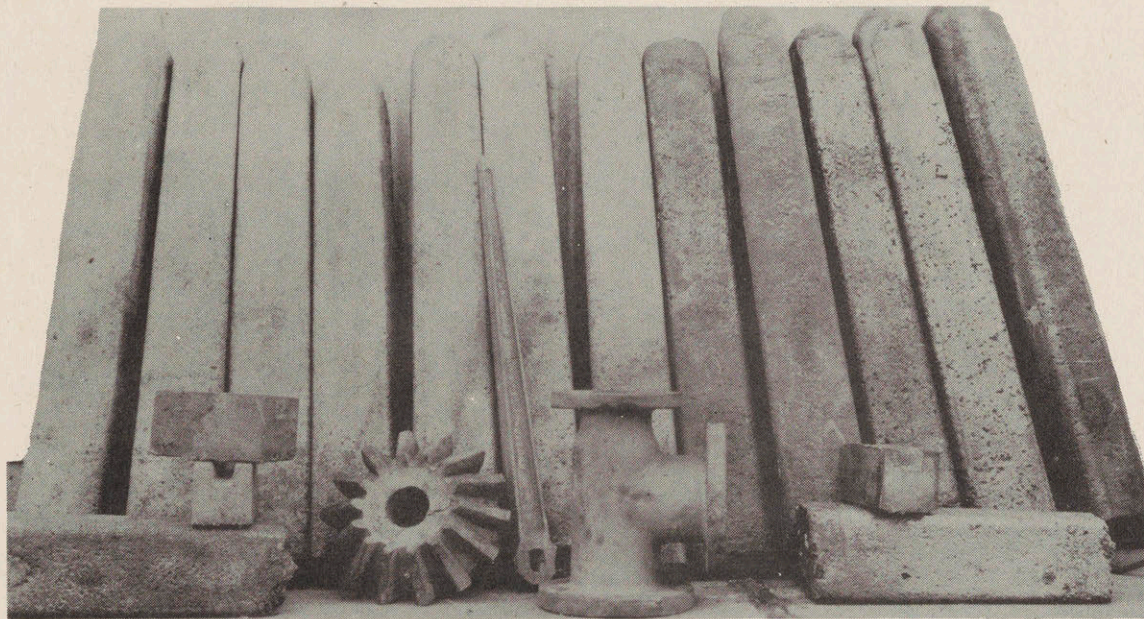


Figure 26. - Some ingots and casting made from sponge iron melted in the electric furnace.

of coke. Materials to withstand the temperature in the surge tank for this method were not available. Iron pipe was used, but it lost its strength and parted at connections, causing leakage and failure of passage of the gas through the coke. Refractory materials were difficult to construct in odd shapes, tended to check and crack, and failed because of leakage.

To determine the effectiveness of the use of coke, the combustion gases from the surge-tank burner must be compared with the same gases after passing through coke. Table 4 shows the composition of the gas when various volumes of air and gas were burned.

TABLE 4. - Gas from combustion of several air - natural-gas mixtures, percent (dry basis)

Quantity of air and gas burned	CO ₂	Illum.	O ₂	H ₂	CO	C ₂ H ₆	CH ₄	N ₂	Surge-tank outlet temp., °F.
200 c.f.m. air, 35 c.f.m. nat. gas.....	6.2	0.1	0.2	11.8	8.2	0.7	0.0	72.8	1,675
200 c.f.m. air, 36 c.f.m. nat. gas.....	5.2	.0	.0	15.2	9.9	.6	.0	69.1	
300 c.f.m. air, 50 c.f.m. nat. gas.....	5.1	.0	.4	13.8	8.2	.9	.0	71.6	1,875
300 c.f.m. air, 54 c.f.m. nat. gas.....	4.7	.1	.3	16.6	10.8	1.0	.0	66.5	
300 c.f.m. air, 60 c.f.m. nat. gas.....	4.3	.1	.1	19.9	10.4	.3	1.6	63.3	

Table 5 shows the result of passing the products of combustion through the bed of coke. By passing these gases through the coke, the carbon dioxide was decreased while the hydrogen and carbon monoxide were increased. These reactions, however, were accompanied by a decrease in temperature.

TABLE 5. - Gas obtained after passing combustion gases through incandescent coke, percent (dry basis)

Quantity of air and gas burned	CO ₂	Illum.	O ₂	H ₂	CO	C ₂ H ₆	CH ₄	N ₂	Surge-tank outlet temp., °F.
200 c.f.m. air, 35 c.f.m. nat. gas.....	4.0	0.0	0.0	17.8	14.9	1.1	0.0	62.2	1,600
300 c.f.m. air, 50 c.f.m. nat. gas.....	3.7	.0	.0	20.0	14.6	1.5	.0	60.2	1,550
100 c.f.m. air, 17 c.f.m. nat. gas.....	4.6	.0	.1	16.7	14.4	.9	.0	63.3	1,560

Table 6 shows the effect of passing generator gas mixed with combustion gas through the bed of coke.

TABLE 6. - Gas obtained after passing both generator gas and combustion gas through incandescent coke, percent (dry basis)

	CO ₂	Illum.	O ₂	H ₂	CO	C ₂ H ₆	CH ₄	N ₂	Surge-tank outlet temp., °F.
(a)									
Generator gas before reaching coke bed.....	1.3	0.0	0.1	32.1	16.6	0.2	7.2	42.5	
Mixture of combustion gas and generator gas after passing through coke bed..	1.7	.1	.1	29.6	16.5	.7	6.2	45.1	1,550
Note: Combustion gas was produced by burning 100 c.f.m. of air with 17 c.f.m. of natural gas. Generator gas, approximately 750 c.f.m. of the composition shown above.									
(b)									
Generator gas before reaching coke bed.....	1.2	0.1	0.4	31.6	16.1	0.1	7.7	42.8	
Mixture of generator gas and combustion gas after passing through coke bed.....	2.2	.0	.4	29.0	15.2	.8	5.5	46.9	1,525
Note: Combustion gas produced by burning 200 c.f.m. of air with 35 c.f.m. of natural gas. Generator gas approximately 750 c.f.m. of the composition shown above.									
(c)									
Generator gas before reaching coke bed.....	1.1	0.0	0.0	31.6	16.8	0.7	7.3	42.5	
Mixture of generator gas and combustion gas after passing through coke bed.....	2.5	.1	.0	26.8	16.8	.6	5.6	47.6	1,540
Note: Combustion gas produced by burning 300 c.f.m. of air with 50 c.f.m. of natural gas. Generator gas, approximately 750 c.f.m. of the composition shown above.									

Table 7 shows the comparison of gas composition when generator gas was mixed with combustion gas not passed through coke, and with combustion gas passed through coke.

TABLE 7. - Comparison of gas mixture not pass through coke and same gas mixture passed through coke

	Analysis, percent (dry basis)							
	CO ₂	Illum.	O ₂	H ₂	CO	C ₂ H ₆	CH ₄	N ₂
Generator gas ^{1/} before reaching coke bed.....	1.4	0.0	0.1	32.5	16.3	0.0	8.0	41.7
Mixed gas ^{2/} not passed through coke.....	2.1	.1	.1	28.6	15.7	.9	5.1	47.4
Mixed gas ^{2/} passed through coke.....	2.0	.1	.1	29.7	16.5	.6	5.1	45.9

^{1/} Generator gas approximately 750 c.f.m. of the composition shown above.

^{2/} Generator gas mixed with products of combustion resulting from burning a mixture of 36 cu. ft. of nat. gas and 200 cu. ft. of air.

A series of experiments was made to determine the gas composition obtainable if generator gas and combustion gas were mixed and not passed through coke. Results are shown in table 8.

TABLE 8. - Effect of mixing generator gas with combustion gas and not passing through incandescent coke

	analysis, percent (dry basis)							
	CO ₂	Illum.	O ₂	H ₂	CO	C ₂ H ₆	CH ₄	N ₂
Generator ¹ /	1.3	0.0	0.0	32.0	16.4	0.2	8.5	41.6
Mixed gas ² /	2.1	.0	.1	26.9	15.4	1.3	4.1	50.1
Generator gas ¹ /	1.5	.0	.1	31.8	16.8	.5	7.9	41.4
Mixed gas ³ /	2.7	.0	.1	29.7	14.3	.8	4.5	47.9
Generator gas ¹ /	1.1	.0	.0	31.9	17.6	1.3	7.0	41.1
Mixed gas ⁴ /	2.1	.0	.0	28.0	16.0	1.0	4.4	48.5

- 1/ Generator gas approximately 750 c.f.m. of the compositions shown above in each case.
- 2/ Generator gas mixed with products of combustion resulting from burning a mixture of 54 c.f.m. of nat. gas and 300 c.f.m. of air.
- 3/ Generator gas mixed with products of combustion resulting from burning a mixture of 60 c.f.m. of nat. gas and 300 c.f.m. of air.
- 4/ Generator gas mixed with products of combustion resulting from burning a mixture of 33 c.f.m. of nat. gas and 200 c.f.m. of air.

Use of Coke for Improving Quality of the Gas

Petroleum coke sized to minus 2-inch plus 3/4-inch was used. In each case the bed of coke was 6 feet in depth at the beginning of the experiment. The volume diminished during the conversion, leaving progressively smaller amounts of effective reducing material. Time of contact of the gases with the coke varied according to the specific test; the greater the volume of combustion gases used, the shorter the contact period. Temperature of the coke and gas at the time of contact is more important than time in the conversion of water and carbon dioxide into hydrogen and carbon monoxide if the contact is efficient. The temperature in the bed during the tests was approximately 1,850° F. Higher temperature would have been desirable for greater conversion, but the following factors were believed to set practical limits:

1. It was doubtful that the equipment available could withstand localized extreme temperatures.
2. A higher temperature required introduction of more combustion gases and hence more nitrogen, which decreased the total percentage of reducing gases by dilution.
3. Too great a velocity of combined combustion gases and generator gases entering the coke bed might cause the light particles of coke to be

suspended in the gas. This would tend to pulverize the coke and cause it to flow with the gas, with possible plugging of the gas line at any of various points.

4. Excess volumes of gas flowing through the coke tended to channel and prevent the desirable gas-coke contact.

5. The greater the gas conversion necessary, as brought about by using more combustion gas, the greater the volume of heat required to sustain the reaction.

A compromise of the desirable and undesirable factors therefore had to be made. After a study of the foregoing tables and a resume of the operating conditions, it was decided that optimum conditions would prevail when the products of combustion from 200 c.f.m. of air and 36 c.f.m. of natural gas were passed through the coke and then mixed with the generator gas.

Run 25. After over-all consideration of these results, run 25 was made with the surge-tank burner set at a rate of 200 cubic feet of air and 36 cubic feet of natural gas a minute and an air:gas ratio of 5.5 to 1. The products of combustion were passed through coke.

Run 26. This run was almost a duplicate of run 25. The reduction was practically the same, but the volume of gas sent to the generator in proportion to the weight of metallic iron produced was slightly lower in run 26 than in run 25. The temperature was slightly higher in run 26 because more gas was burned in the surge tank.

In both runs 25 and 26, the combustion gases and reducer gases were passed through the coke.

Run 27. Run 27 was started with the intention of passing only the combustion gases through the coke. After 7 hours 35 minutes of operation, shutting down was necessary because of mechanical troubles with the air compressor. Analysis of the sponge showed 79.4-percent reduction in the reducer bottom and 37.2-percent at the top of the charge. Samples taken at intermediate points were reduced in proportion to their relative position in the chamber.

Run 28. This run failed because the coke was blown into the line leading from the surge tank to the reducer and shut off the flow of gas.

Run 29. Catalyst instead of coke was used in the surge tank. The ore was heated with a reducing gas. The temperatures in the surge tank kept increasing until it was feared that the thermocouples would burn out.

It was thought that increasing the volume of gas might cause excess carbon deposition on the catalyst and ore and that diminishing both air and gas would lower the heat input to the point where no heat transfer to the ore could be effected. Finally a large excess of air was used, and this also resulted in continued rise of temperature. The original air:gas ratio

was set at 6 : 1, and heating was continued until the ore temperature reached 1,900° F. Meanwhile the bottom-zone thermocouple in the reducer had broken because of the high temperature and the burden of the ore charge. Although the reduction proceeded without apparent trouble and high reduction temperatures were maintained, it was later found that the ore had fused in the reducer bottom when the excess air was used to cool the surge tank, leaving only a small channel through the center. Consequently, the data were not informative.

Run 30. The final run 30 was made on beneficiated Minnesota ore which had been converted into nodules of minus 2-inch plus 1/4-inch size. The ore was heated to nearly 2,000° F. before the reducing gas was introduced into the reducer. Again the generators failed to function properly. In an effort to obtain some data of value, the surge-tank burner was adjusted to a 2.50 : 1 air:gas ratio and used as a generator without the benefit of preheated air and gas.

Average reducing-gas analysis during run 30

<u>Gas</u>	<u>Percent by volume</u>
H ₂ O	4.0
CO ₂	2.8
Illum.	.7
O ₂	.4
H ₂	14.7
CO	10.2
C ₂ H ₆	.4
CH ₄	18.0
N ₂	48.8

Average temperature during run 30, °F.

Surge-tank outlet....	1,806
Bottom zone.....	1,364
Middle zone.....	1,402
Top zone.....	1,380

The duration of the test was 15.5 hours. Removal of the ore was very difficult as it had to be removed manually by hoisting out through the top of the reducer. Particles on the top of the charge showed virtually no reduction but the reduction increased to 82.7 percent in the bottom. Sticking was caused by the high preheat temperature before reduction.

Reduction Reactions

Calculations for reduction reactions can be made only if several assumptions are accepted.

The temperature of the reducing gas was known to be higher than the temperature of the ore. The temperature of the ore was highest in the

bottom zone followed by the middle zone. The temperature of the top of the ore was not known with accuracy since the ore generally shrank during reduction and the top of the charge dropped to about 6 feet below its original level, thus leaving the top thermocouple above the ore.

A close approximation of the true ore temperature may be calculated from the average of:

1. Surge-tank outlet
2. Reducer bottom zone
3. Reducer middle zone
4. Reducer top zone

Another assumption is that the temperature of any given zone can be averaged over the length of a run. Since the ore temperature was always higher at the beginning of a test than at any subsequent time, the assumption that an average can be taken is conservative.

Finally the assumption that the quality of the reducing gas remained constant throughout the test must be made.

Therefore the temperature of the ore and the analysis of the gas used in the following calculations are the averages obtained from periodical observations during run 26, which are summarized as follows:

Calculations concerning total time required to reduce
iron oxide to metallic iron

(Gas analysis and temperature taken from run 26)

<u>Reducing gas</u>	<u>Percent by volume</u>
H ₂ O.....	3.6
CO ₂	2.1
O ₂	0.2
H ₂	26.5
CO.....	15.6
CH ₄	7.1
N ₂	<u>44.9</u>
Total.....	100.0

Ore, 25,000 lb.

Iron content, 50 percent = 12,500 lb. iron

Re-formed gas, 784 cu. ft. a minute

Re-forming conditions, 160 cu. ft. natural gas and
400 cu. ft. air a minute

Volume of re-formed gas, 1.4 combined volumes of
natural gas and air

Duration of run, 960 minutes (16 hours)

Average temperature, 1560° F.

Data from:

American Gas Association, Combustion: New York, 1932, 208 pp.

Emmett, P. H., and Shultz, J. F., Influence of Gaseous Thermal Diffusion on Equilibrium Measurements of the Fe-O-H System: Jour. Am. Chem. Soc., vol. 54, 1932, pp. 3780-3781.

Eastman, E. C., and Evans, R. M., Equilibria Involving Oxides of Iron: Jour. Am. Chem. Soc., vol. 46, 1924, pp. 888-903.

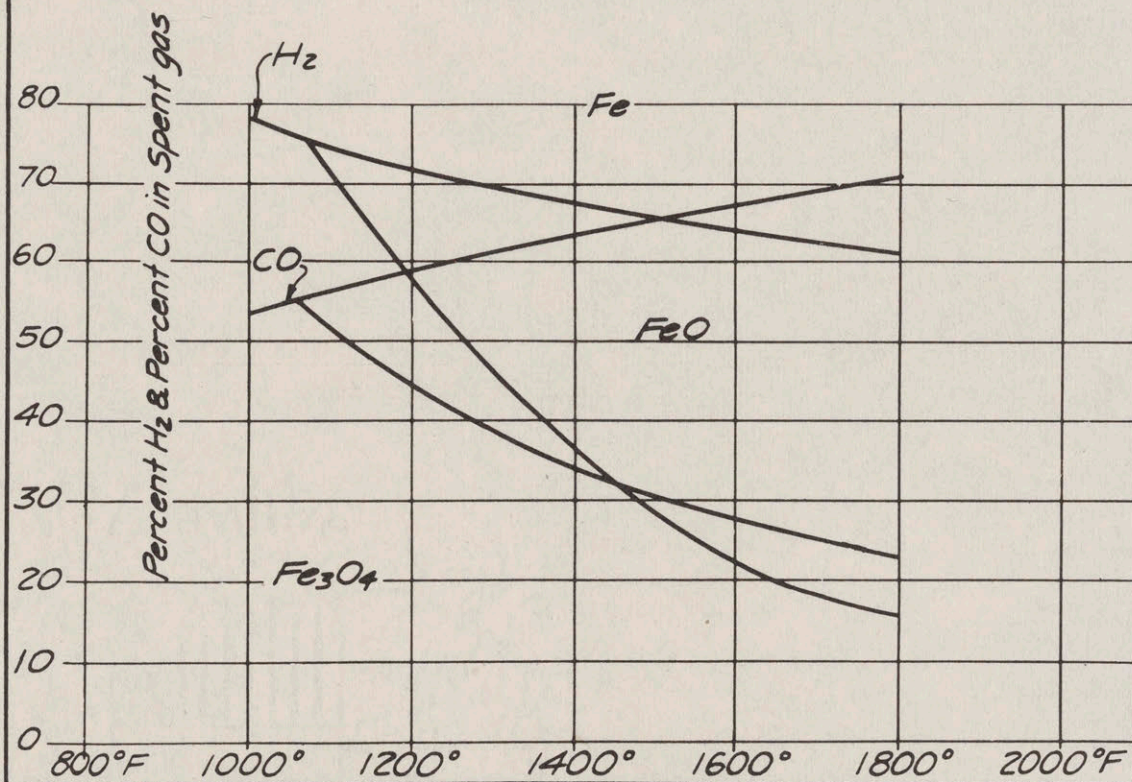


FIGURE 17.- Hydrogen and carbon monoxide equilibria with iron and iron oxides.

$K_1 = \frac{H_2O}{H_2}$ (FeO-Fe): See American Gas Association, Combustion: New York, 1932, 208 pp.

$K_2 = \frac{CO_2}{CO}$ (FeO-Fe): See above reference.

$K_3 = \frac{H_2O}{H_2}$ (Fe_3O_4 -FeO): See Emmett, P. H., and Shultz, J. F., Influence of Gaseous Thermal Diffusion on Equilibrium Measurements of the Fe-O-H System: Jour. Am. Chem. Soc., vol. 54, 1932, pp. 3780-3781.

$K_4 = \frac{CO_2}{CO}$ (Fe_3O_4 -FeO): See Eastman, E. C., and Evans, R. M., Equilibria Involving Oxides of Iron: Jour. Am. Chem. Soc., vol. 46, 1924, pp. 888-903.

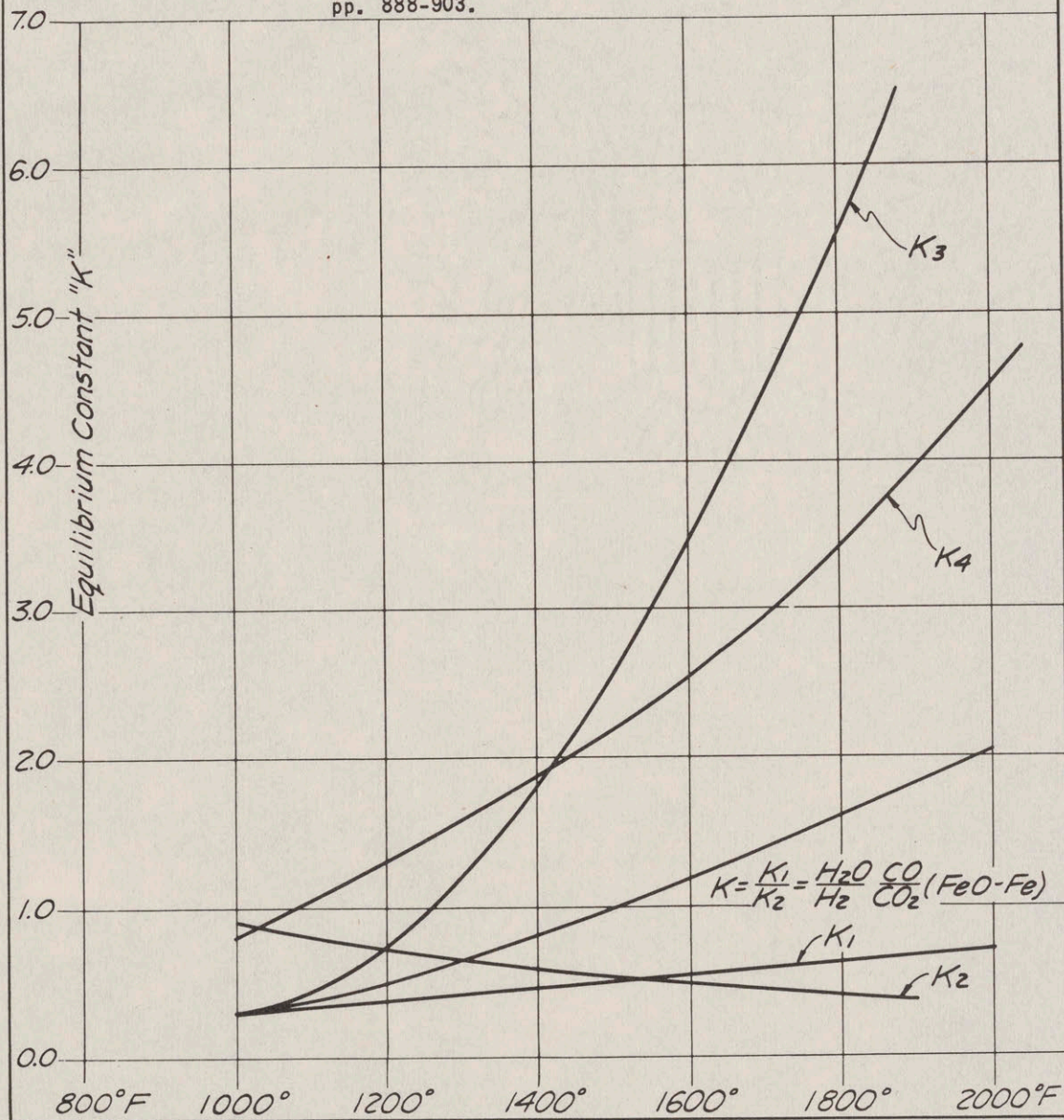


FIGURE 18.- Equilibrium constants.

Calculations are based on reduction in two stages:

The curves of equilibrium in figures 17 and 18 show that the ratios of H_2O to H_2 and CO_2 to CO in gas in contact with iron oxide will be respectively 3 and 2.4.

$$(Fe_3O_4 - FeO), K_3 = \frac{H_2O}{H_2} = 3.00, K_4 = \frac{CO_2}{CO} = 2.4$$

The gas from the generator already contains these constituents and it will be changed in its passage through the ore, by decrease of H_2 and increase of H_2O and decrease of CO and increase of CO_2 , to such extent that these constituents will be present in the ratios given above.

$$\text{For } K_3 \quad \frac{3.6 + x}{26.5 - x} = 3.0, x = 19.0$$

$$\frac{3.6 + 19.0}{26.5 - 19.0} = \frac{22.6}{7.5} = 3.0$$

$$\text{For } K_4 \quad \frac{2.1 + x}{15.6 - x} = 2.4, x = 10.4$$

$$\frac{2.1 + 10.4}{15.6 - 10.4} = \frac{12.5}{5.2} = 2.4$$

At equilibrium the composition of the gas will be theoretically:

	Percent
H_2O	22.6
CO_2	12.5
O_2	.2
H_2	7.5
CO	5.2
CH_4	7.1
N_2	44.9
	100.0

Theoretically, this composition would prevail if the ore was reduced uniformly. This is not the case, however, since the ore in the bottom of the reducer is probably partly reduced to metallic iron, while Fe_3O_4 and FeO in some proportion may still exist in the upper part of the reducer. Since all the Fe_3O_4 must first go through the FeO state at some period during the reduction, the above calculations are probably correct for the first few minutes of the reduction. As the reduction proceeds into the FeO to Fe stage, part of the first gas reactions will not have been completed and will lag into the second reaction. Thus the over-all average will compensate and two-phase calculations are justified:

$\text{Fe}_3\text{O}_4 = 27.6$ percent oxygen 72.4 percent iron

$\text{FeO} = 22.2$ percent oxygen 77.8 percent iron

100 lb. Fe is equivalent to $(100 \div 0.724) = 138$ lb. Fe_3O_4

100 lb. Fe is equivalent to $(100 \div 0.778) = 129$ lb. FeO

Considering 100 lb. of iron:

$138 - 129 = 9$ lb. oxygen must be removed in the Fe_3O_4 to FeO conversion. The total amount of oxygen to be removed to convert the entire charge from Fe_3O_4 to FeO is:

$\frac{12,500}{100} \times 9 = 1,125$ lb. The volume of 1 pound of dry oxygen at 60° F. and 30 inches mercury pressure is 11.82 cu. ft.^{13/}

$1,125 \times 11.82 = 13,297$ cu. ft. of oxygen to be removed.

The calculation of equilibrium shows that 19 percent of the gas was effective in the reduction in the form of hydrogen and 10.4 percent in the form of carbon monoxide or a total of 29.4 cu. ft. out of every 100 cu. ft. of reducing gas is completely utilized.

Hence the gas utilized is $784 \times \frac{29.4}{100} = 230$ cu. ft. a minute. The reactions ($\text{H}_2 + 1/2 \text{O}_2 = \text{H}_2\text{O}$, and $\text{CO} + 1/2 \text{O}_2 = \text{CO}_2$) show that each cubic foot of reducing gas utilized removes 1/2 cu. ft. of oxygen. Therefore 230 cu. ft. of reducing gas removes 115 cu. ft. of oxygen. The time required for the removal of oxygen in the reduction of Fe_3O_4 to FeO will be:

$$\frac{13,297}{105} = 116 \text{ minutes} = 1 \text{ hour } 56 \text{ minutes}$$

The curves of equilibrium show that the ratios of H_2O to H_2 and of CO_2 to CO for reduction of FeO to Fe will be:

$$K_1 = \frac{\text{H}_2\text{O}}{\text{H}_2} = 0.5$$

$$K_2 = \frac{\text{CO}_2}{\text{CO}} = 0.5$$

$$\text{For } K_1, \quad \frac{3.6 + x}{26.5 - x} = 0.5, \quad x = 6.4$$

$$\frac{3.6 + 6.4}{26.5 - 6.4} = \frac{10.0}{20.1}$$

^{13/} Work cited in footnote 5, p. 100.

$$\text{For } K_2, \frac{2.1 + x}{15.6 - x} = 0.5, x = 3.8$$

$$\frac{2.1 + 3.8}{15.6 - 3.8} = \frac{5.9}{11.8}$$

At equilibrium the composition of the gas will be theoretically:

	<u>Percent</u>
H ₂ O	10.0
CO ₂	5.9
O ₂	.2
H ₂	20.1
CO	11.8
CH ₄	7.1
N ₂	44.9

The total volume of oxygen to be removed in the reduction of FeO to Fe is:

$$\frac{12,500 \times 29 \times 11.82}{100} = 42,847 \text{ cu. ft.}$$

Of each 100 cu. ft. of gas, 6.4 + 3.8 = 10.2 cu. ft. is utilized in the reaction. The volume of gas utilized a minute is:

$$\frac{784 \times 10.2}{100} = 80 \text{ cu. ft.}$$

Each cubic foot of gas utilized removes 1/2 cu. ft. of oxygen, therefore the time required for complete reduction of the ore, assuming this to be possible, is:

$$\frac{42,847}{40} = 1,070 \text{ minutes} = 17 \text{ hours } 51 \text{ minutes}$$

Total time required is:

(Fe ₃ O ₄ - FeO)	1 hour 56 minutes
(FeO - Fe)	<u>17 hours 51 minutes</u>
	19 hours 47 minutes

Since 19.8 hours was theoretically required for complete reduction, 16 hours should show $\frac{16}{19.8} = 81$ percent reduction. This extent of reduction

was very nearly attained as analysis of the reduced ore from this particular test showed 80.0 percent reduction. This assumes that time is directly proportional to reduction which is not exactly the case. Since the calculations have been based on both stages of the reactions proceeding at the same time, this calculation is permissible.

Under the conditions discussed above, run 26 should have required 30,341 cu. ft. of natural gas for reduction of each ton of metallic iron produced, as follows:

R.I. 3925

$$\frac{12,500}{2,000} = 6.25 \text{ tons of Fe in charge,}$$

$$160 \times 960 = 153,600 \text{ cu. ft. of natural gas used.}$$

Reduction of iron oxides to metallic iron = 81 percent

$$6.25 \times 0.81 = 5.0625 \text{ tons of iron produced in metallic form.}$$

$$\frac{153,600}{5.0625} = 30,341 \text{ cu. ft. of natural gas used a ton of iron produced.}$$

The actual amount of gas used a ton of metallic iron, not including that used in the surge tank burner, was 31,425 cu. ft.

Up to this point (16 hours reduction time) the actual results correspond fairly closely with the theoretical calculations. Beyond this time, with the reduction temperature and low quality gas available, it is believed that the rate of reaction becomes so small that an impracticable length of time would be required to complete the reduction.

Equilibrium curves are not developed using time as a factor, and equilibrium may not be established during the latter part of the reduction. Actual gas analyses support this supposition.

The rate of reaction during run 26 is shown in figure 19. Also shown are reaction rate curves (figs. 20, 21, and 22) for other tests with various degrees of reduction. Percentages of carbon dioxide and water vapor are shown at any given instant during the test. The reacted gas is the percentage in the spent gas minus the percentage in the make gas. Extensions of these curves indicated by the broken lines are rates projected to satisfy the foregoing calculations. The curves probably become asymptotic after 16 hours. As mentioned previously, carbon monoxide tended to decompose toward the end of the test, and the formation of carbon dioxide retarded the reaction rates considerably.

The total heat of reaction in early tests was endothermic, while in the later tests conditions were sufficiently exothermic to maintain a fairly steady temperature despite radiation losses. Although calculations for a thermal reaction balance are not justified by the conditions of the test, the following ore-gas thermal balance is calculated from data of run 26 for the sake of completeness and theory.

The gases entering into the equilibrium have the following partial percentage analysis:

H ₂ O	3.6
CO ₂	2.1
H ₂	26.5
CO	15.6

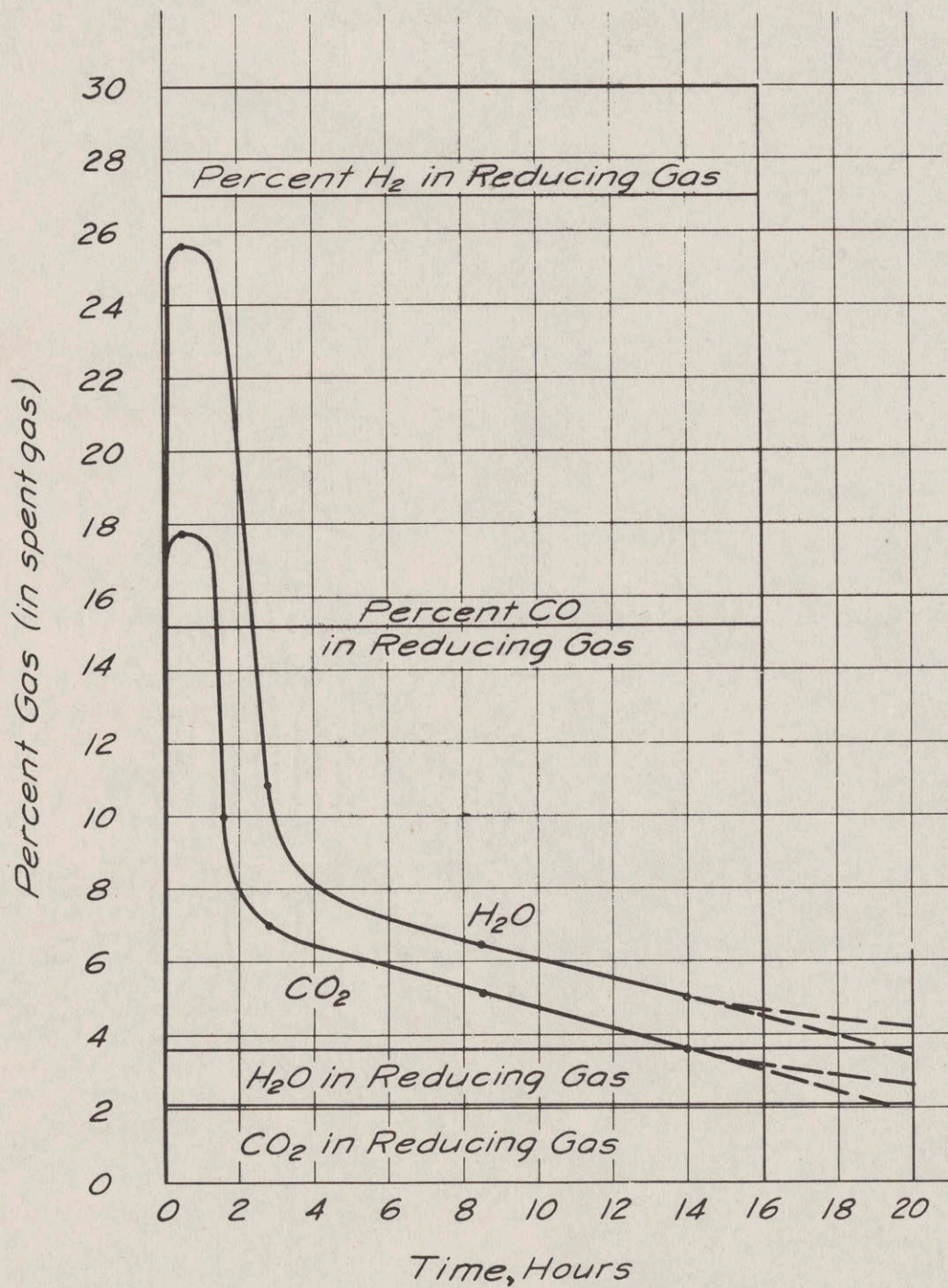


FIGURE 19.- Reduction test 26.

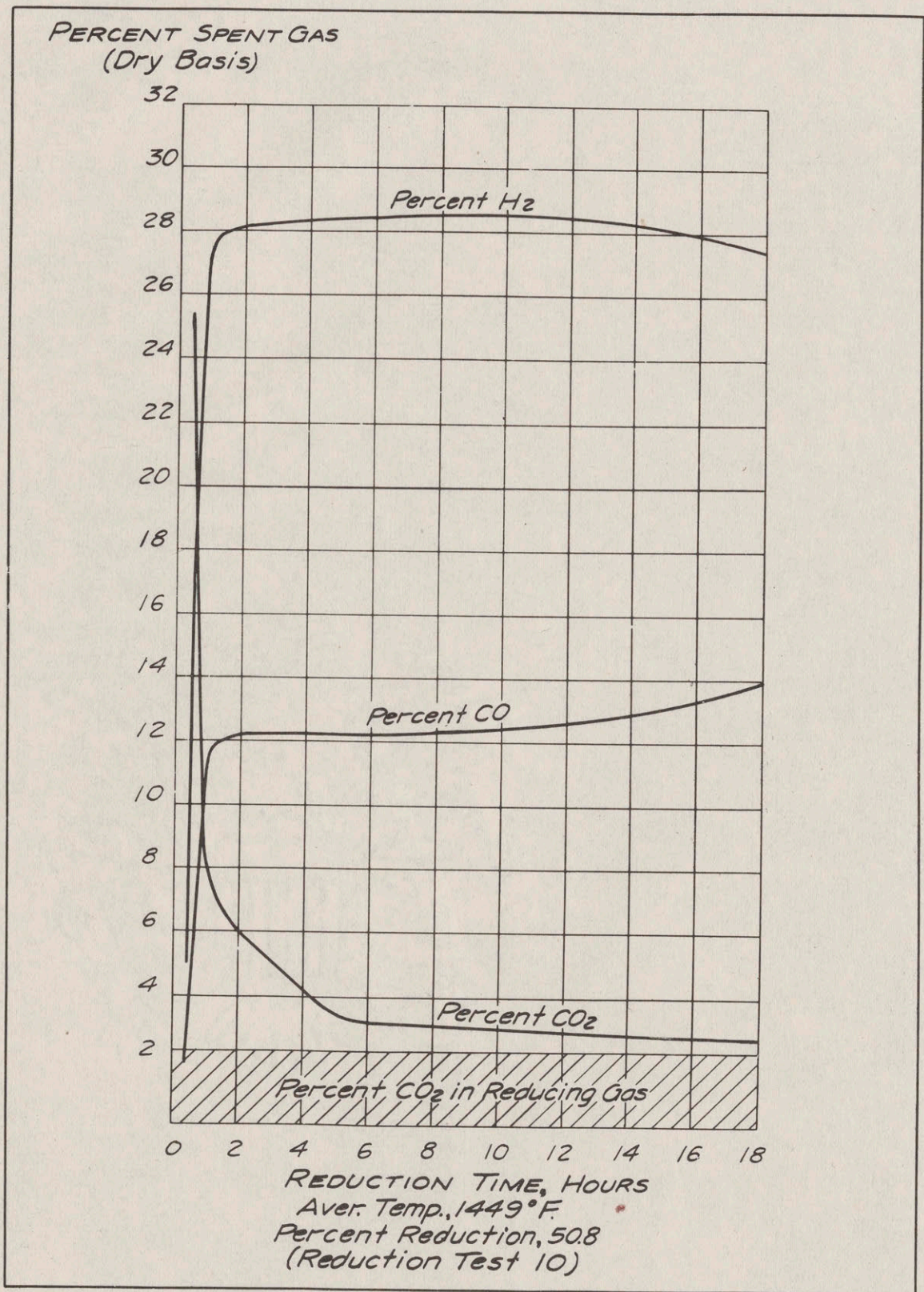


FIGURE 20.- Spent-gas composition vs. reduction time.

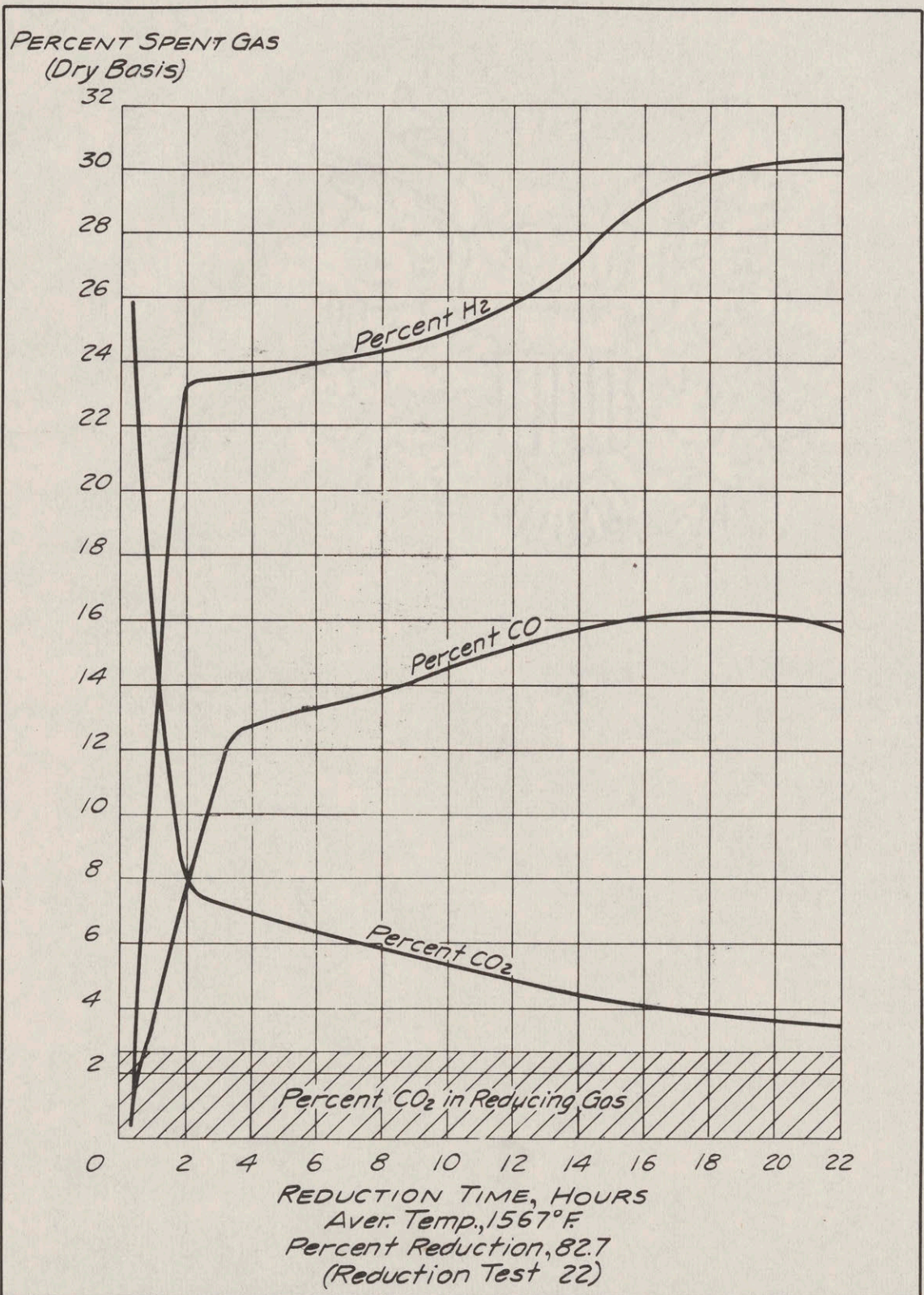


FIGURE 21.- Spent-gas composition vs. reduction time.

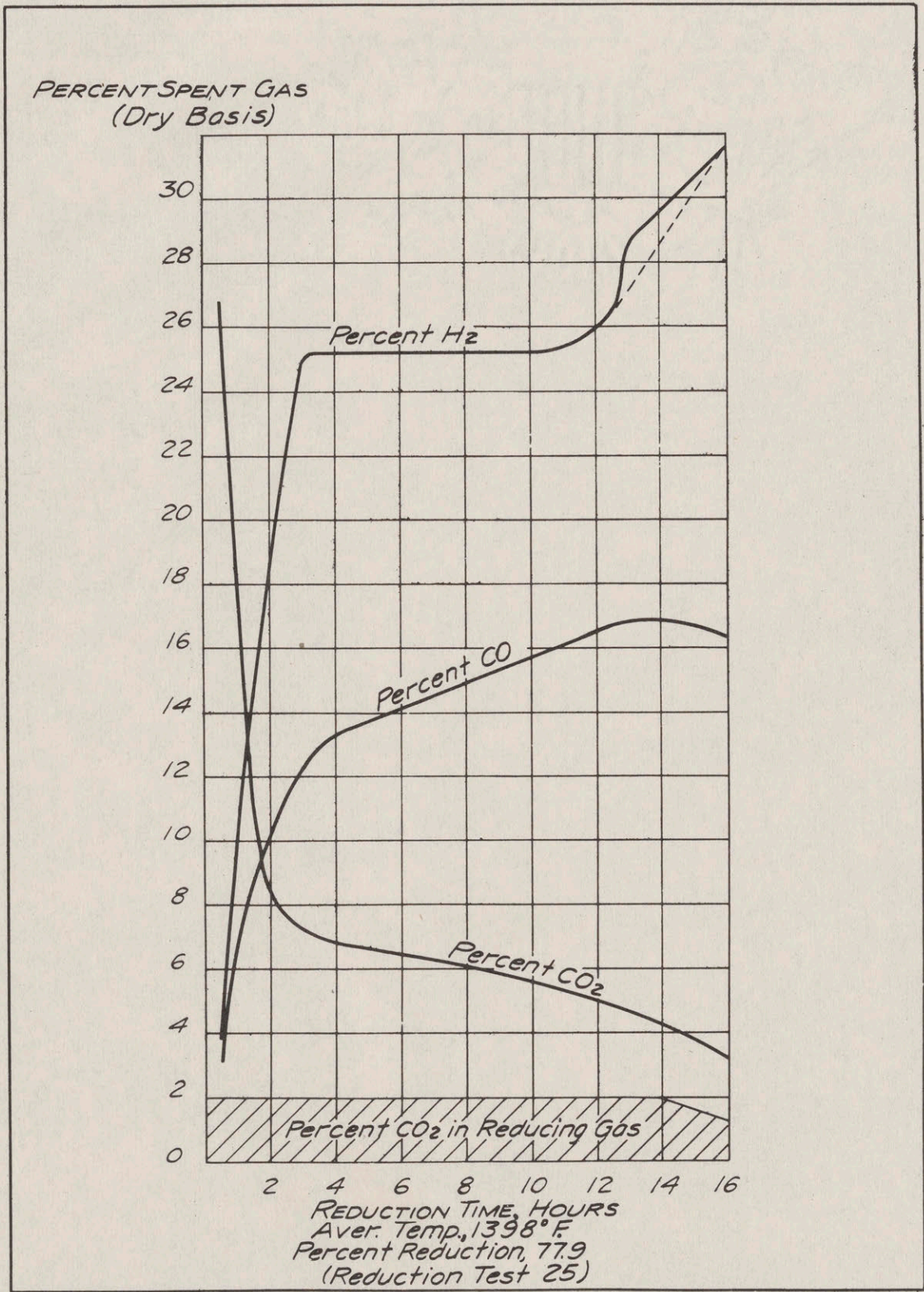


FIGURE 22.- Spent-gas composition vs. reduction time.

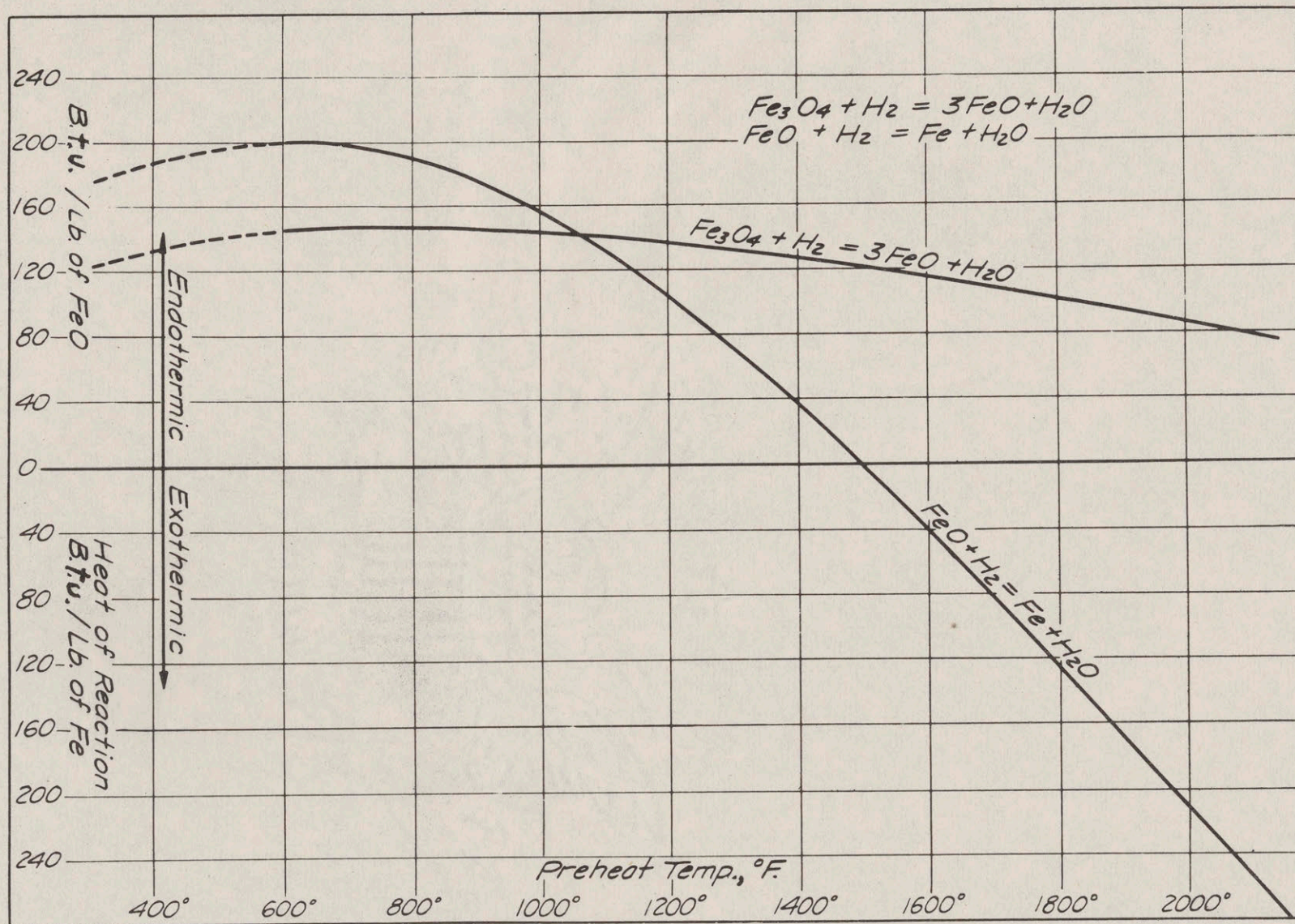


FIGURE 23.- Reaction heats vs. preheat of reagents.

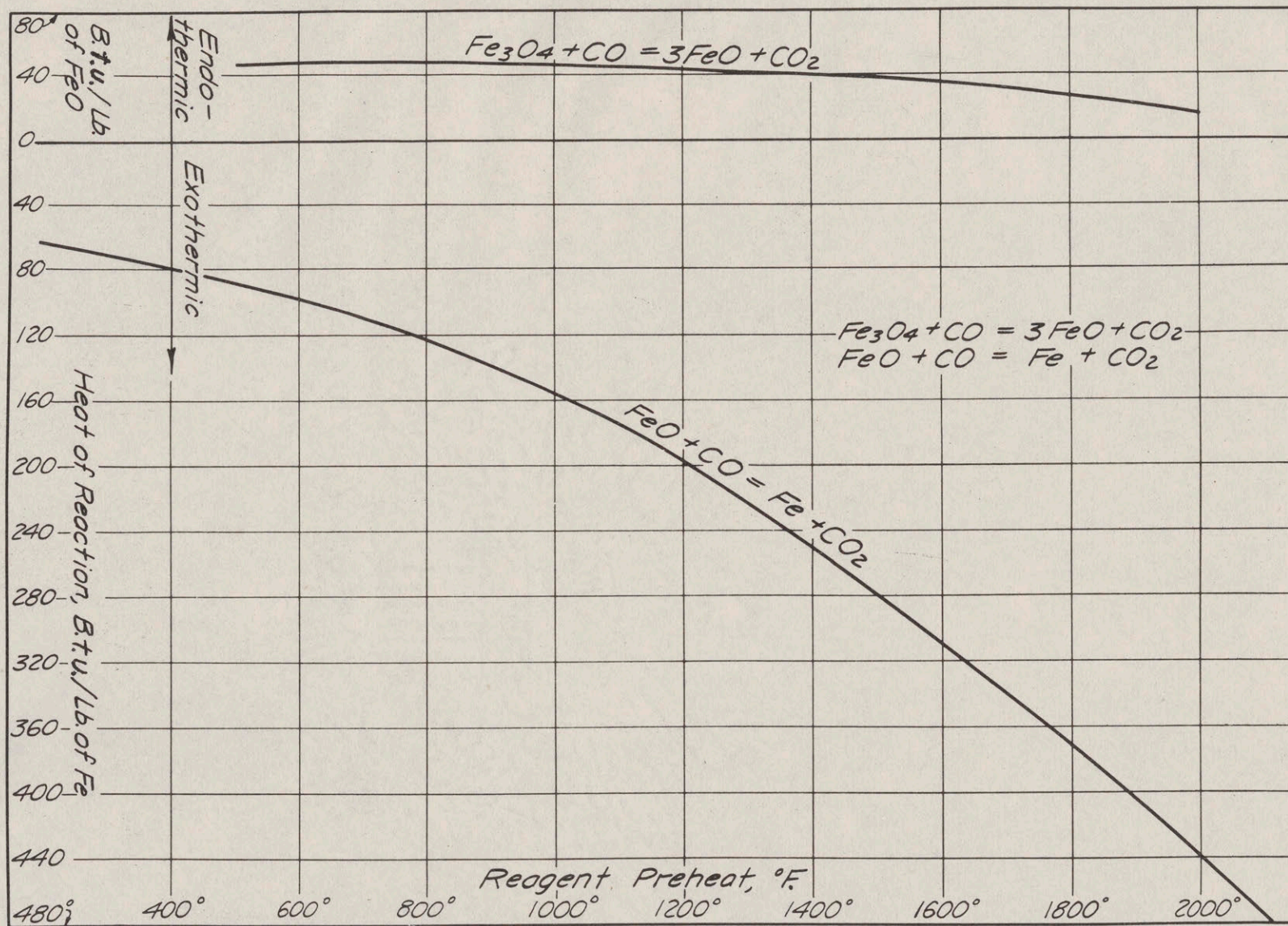


FIGURE 24.- Reaction heats vs. preheat of reagents.

At 1,560° F. and under equilibrium conditions, 75 percent of the hydrogen and 71 percent of the carbon monoxide will have been consumed to produce additional water and carbon dioxide, respectively. The partial percentage analysis of the gases at equilibrium will be:

H ₂ O	23.475
CO ₂	13.176
H ₂	6.625
CO	4.524

The heats of reaction (figs. 23 and 24) at 1,560° F. are -120 B.t.u. a pound of FeO for the hydrogen reduction and -35 B.t.u. a pound of FeO for the carbon monoxide reduction.

The above tabulated analyses show that 19.875 percentage units of hydrogen and 11.076 percentage units of carbon monoxide are consumed by reduction of Fe₃O₄ to FeO. Hydrogen therefore will perform 64.21 percent of the reduction and carbon monoxide will perform the other 35.79 percent of the reduction.

The heat consumption incurred when producing 1 pound of FeO by reduction with the gas mixture will be:

$$\begin{array}{r}
 0.6421 \times -120 = -77.05 \\
 0.3579 \times -35 = -12.53 \\
 \text{Total} \qquad \qquad -89.58 \text{ B.t.u. a pound of FeO}
 \end{array}$$

Obviously, the first stages of reduction are strongly endothermic.

Similar calculations may be made for the second phase of the reduction. From the heat-of-reaction graphs it is seen that the FeO to Fe reaction with hydrogen is exothermic to the extent of 25 B.t.u. a pound of iron produced. The carbon monoxide reaction is exothermic at 1,560° F. and 295 B.t.u. a pound of iron are developed. The equilibria diagram indicates that 65 percent of the hydrogen and 67 percent of the carbon monoxide available will be in the spent gas.

It may be assumed that the two reactions, (a) Fe₃O₄ = 3 FeO + O and (b) FeO = Fe + O, are progressing at the same time, and that the second reaction may be considered under the same equilibrium conditions as the first.

The partial percentage analysis of the gases at equilibrium for the second reaction therefore will be:

H ₂ O	12.875
CO ₂	7.248
H ₂	17.225
CO	10.452

Comparison of the above tabulation with the partial analysis of gases entering into equilibrium shows that 9.275 percentage units of hydrogen and 5.148 percentage units of carbon monoxide are consumed by reduction of FeO to Fe. Hydrogen therefore will perform 64.3 percent of the reduction and carbon monoxide will perform the other 35.7 percent of the reduction.

The heat generated when producing 1 pound of iron by reduction of FeO with the gas mixture will be:

$$\begin{array}{r} 0.643 \times 25 = 16.075 \\ 0.357 \times 295 = \underline{105.315} \\ \text{Total} \quad \quad 121.39 \text{ B.t.u. a pound of Fe} \end{array}$$

The exothermic heat produced by this second reaction is for 1 pound of iron and is equivalent to 94.41 B.t.u. a pound of FeO.

The over-all heat effect of reducing Fe_2O_4 to Fe is exothermic, and its value is found to be 4.83 B.t.u. a pound of FeO or 6.21 B.t.u. a pound of iron (by algebraic addition of the heat effects for the two reactions).

SIGNIFICANCE OF DATA AND OBSERVATION WITH RESPECT TO ESTABLISHED FACTS

The production of reducing gas was not entirely successful in that the water vapor and carbon dioxide contents were too high for good reduction. Continuous operation of the generators was not accomplished. The temperature of the reducing gas was too low for direct use in the reduction system. Unless more time can be spent on the development of gas re-forming, some commercial type of generator with well-established characteristics will be needed. The presence of approximately 50 percent inert nitrogen in the gas increased the length of time necessary to obtain adequate reduction of the ore.

The loss of heat by radiation was very great. The replacement of lost heat by burning natural gas directly into the reducing gas did not solve the problem but provided enough heat to obtain 80 percent reduction before the generators would fail. The plant had not been designed with heat conservation in mind, and the heat losses were still excessive after a considerable amount of additional insulation was installed. In the design of a large plant, extreme emphasis must be placed on heat conservation.

Preheating of the ore to above $1,900^\circ \text{F}$. before reduction generally caused sticking of the charge in the reducer and discharging was difficult. The design of the ore reducer should be such that the inside would have a smaller diameter at the top than at the bottom. This would necessitate the installation of a specially designed pressure valve with an opening equal to that of the bottom of the reducer.

A neutral or reducing atmosphere must be available to protect the hot sponge. Cooling systems must be designed to cool the sponge and, unless it can be disposed of immediately, a very dry storage system must be provided.

A pile about 4 feet in diameter and 2 feet in thickness had been set to one side of the ground. This material was about 75 percent reduced and was about 2 weeks old when a fine misty rain fell. When a dense vapor was noticed rising from the pile, some heat measurements were made in holes of different depths. Air temperature was 41° F.

<u>Hole No.</u>	<u>Depth below surface, inches</u>	<u>Temperature, °F.</u>
1.....	2.75	160
1.....	3.50	169
2.....	2.75	163
3.....	2.75	193
3.....	3.50	203
3.....	4.25	204
3.....	5.00	201
3.....	5.75	194

Two gas generators at Longview produced a total of 1,570 cubic feet of reducing gas a minute expressed at standard conditions. With the addition of the combustion gases of the surge-tank burner, about 2,000 cubic feet a minute was actually available.

When using one gas generator and pulsations at the rate of three a minute, 333 cubic feet of gas would be admitted to the ore with each blast. As soon as the outlet valve was opened the pressure was relieved and the gas expanded. Enough gas was admitted under these conditions to cause lifting of the ore if less than 20,000 pounds was originally charged. The lifting effect was not noticed when the pulsating valves were not used. Any future plant design should provide for elimination of this factor.

Removal of the fine sizes from the ore charge is important because of the possibility of plugging of the gas inlet.

When ore sized to minus 1-inch plus 5/32-inch was used and both lower and upper reducers were charged, the resistance was too great for satisfactory circulation of gas with the equipment available.

The temperature of the gas leaving the lower reducer was not high enough to preheat a charge in the upper reducer. Provisions for burning the spent gas, which had considerable combustible content, in the upper reducer would have to be made to obtain a temperature high enough for immediate reduction after the lower charge was completed.

Since the ore was not removed as it was reduced, the most efficient utilization of the reducing gas was not possible. Reduction in the bottom of the reducer would be completed before reduction in the top was more than about 50 percent complete. The gas in the upper reducer should be used for economy.

Before the sponge iron was discharged, the system had to be purged with neutral gas to eliminate danger of explosion. Ordinarily the sponge iron was left in the reducer at the end of a test, and natural gas was passed through it until it was cool enough to discharge, or below 400° F. Despite these precautions, the very fine material discharged often heated spontaneously.

The tests definitely showed that ore sintered with coke and water at a high temperature was inferior for reduction purposes because of the dense, glazed surface of the sinter.

The operating time required to produce sponge iron 80 percent reduced seemed to be about 13 hours. The actual time of operation depends partly on the quantity of ore charged. If 25,000 pounds of ore containing 12,500 pounds of available iron was charged and the reduction was 80 percent, 1 ton of metallic iron was produced in 2.6 hours. The average amount of natural gas used in 13 hours was 190,314 cubic feet or 38,063 cubic feet a ton of metallic iron. The amount of gas actually used in the reduction reaction would be a smaller figure, perhaps approximately 25,000 cubic feet.

In the gas-re-forming operation about 160 cubic feet of natural gas a minute was used. After mixture with air, and allowing for an increase of volume to 1.4 times the sum of the volumes of air and gas, approximately 784 cubic feet of reducing gas was produced each minute. Therefore 25,000 cubic feet of natural gas produces 125,000 cubic feet of re-formed gas. About 55 percent of this gas is not of a reducing nature, so 56,250 cubic feet of combined carbon monoxide and hydrogen was required in the reduction reaction for each ton of metallic iron produced. Natural gas in Longview was supplied for 5 cents a thousand cubic feet. The cost for gas actually entering the reduction was about \$1.28 a ton of metallic iron produced. Addition of the cost of gas used for preheating and for the surge tank burner makes the total cost for gas approximately \$1.90.

FACTORS AFFECTING OPERATION NOT DEFINITELY EVALUATED

More data on reduction would have been available if a reducing gas of better quality had been obtained. Passing the gas through incandescent coke did not improve it sufficiently to give satisfactory data.

The actual temperature can be approximated by computation from known conditions,^{14/} and in one instance in which this method was applied it was found that the recorded temperature was 92 degrees lower than the computed temperature. The true gas temperature may not be recorded when the temperature of the walls of the duct through which the gas flows is not the same as that of the gas. Since no external heat is applied, the wall temperature is undoubtedly lower than the gas temperature. On the whole, it is believed that the temperatures recorded were 50° to 100° lower than the actual temperatures. The temperatures given in this report, however, are those registered by the instruments.

^{14/} Work cited in footnote 5, p. 560.

Much controversy has arisen concerning the value of pressure in a system of gas and solids. This effect was not truly evaluated during the program.

Two reduction tests, one with only enough pressure to overcome the resistance of the ore and the other with pressure maintained by adjustment of the spent-gas outlet valve, produced approximately the same quality of sponge. The pulsating mechanism was not used in these tests. In a third test in which the pulsating valves were used, no appreciable benefit was found on either the rate or the extent of reduction. Results of two or three tests are not conclusive, and further investigation should be made. The first cost of a plant operated under pressure will be high. Actual operating problems will be increased and plant flexibility limited.

The time required for complete reduction was not determined. Technically this time should be known, but actually in commercial operation its determination not be justified. Enough carbon is deposited on the sponge during the reduction to complete the reduction of residual oxides if the product is ultimately to be melted.

Ores from different regions and of different types may have special characteristics. East Texas ore was used in all cases except one. The one test on Minnesota nodules was made under adverse conditions and could not be properly evaluated. Sintered ore may react similarly to some of the more dense magnetites which are known to be reduced slowly.

In the earlier reduction tests, the net heat of reaction was known to be endothermic, since the ore temperatures diminished gradually until too low for any practical reduction. Later tests showed exothermic reaction after conversion of the ore from the Fe_3O_4 to the FeO stage, as the temperature during the FeO reduction stage was well-maintained.

CHANGES IN PLANT DESIGN TO ALLOW COMMERCIAL OR SEMICOMMERCIAL OPERATION

The cost of operation was not determined in the testing program. Much time was spent in redesigning and altering various parts of the plant, and the actual costs would not have indicated the cost of a commercial operation. It is believed also that changes in design would have been desirable to obtain more satisfactory operation.

To determine the changes necessary and the cost of operation allowable, an outlet for the product at a fairly definite price should be established. Because of the high phosphorus content of the material, use in an electric furnace does not seem practical, and no other market is known. For these reasons the following discussion is based on consideration of the factors that will assure satisfactory operation rather than on those that will permit production for a known market at a definite price.

Assuming the retention of two of the principal features of the Madaras process - use of reducing gas under pressure and pulsating feed of the gas -

certain changes in operation will be necessary. The quality of the reducing gas must be improved.

In the process that has been tested, natural gas is partly burned with air to produce carbon monoxide and hydrogen. This reaction is exothermic but not highly so. The quality of the reducing gas is impaired by the large volume of nitrogen added with the air. It is believed that better results will be attained by a reaction between natural gas and steam to produce carbon monoxide and hydrogen. The carbon dioxide produced and a large part of the unreacted water vapor can be removed and a reducing gas prepared that will consist essentially of the desirable gases, carbon monoxide and hydrogen. Although oxidation of natural gas with steam is a strongly endothermic reaction, the low price of natural gas as a source of heat may make the process practical.

Another change of importance would be redesigning of the discharge system of the reducer. A means would also have to be provided to cool large quantities or reduced iron quickly after discharging.

A schematic diagram for a more suitable operation is shown in figure 25.

Natural gas and steam are mixed and delivered to a generator, which produces hydrogen and carbon monoxide in volumetric ratio of 3 : 1, respectively. Residual water and carbon dioxide are removed in cooling and scrubbing towers, and the gas passes to a receiver. After compression, the gas is stored in a large gas holder. If the pulsating valves are to be used, one of them will be placed on the outlet side of the gas holder. In the original plant design, the pulsating valve was in the hot-gas line and had to be water-cooled with resultant loss of heat. Two heat-resisting alloy coils are provided to preheat the gas to reduction temperature. The gas may be preheated in both coils at once or in only one at a time. Duplicate coils should be used to allow for repairs to either. The gas is admitted to the reducer bottom through well-insulated pipe. The other pulsating valve is installed on the outlet side of the reducer. Either before or after reaching the valve, the line is tapped to withdraw part of the spent gases, which have an appreciable amount of unused combustible gases after the reduction passes the Fe_3O_4 to FeO stage. This gas may be used in several places. If necessary, it can be enriched with natural gas for use at the boiler or gas-preheating coils unit. A part of the spent gas may be directed to the cooling and scrubbing towers and reused. The remaining part could be burned with air in the upper reducer to preheat the next ore charge.

Shown below the reducer are the cars for receiving the finished sponge iron. Receiving cars may be designed so that the ore may be cooled with natural gas. In turn, gas leaving the cars should be stored for reuse or enriched with natural gas and burned at the boiler.

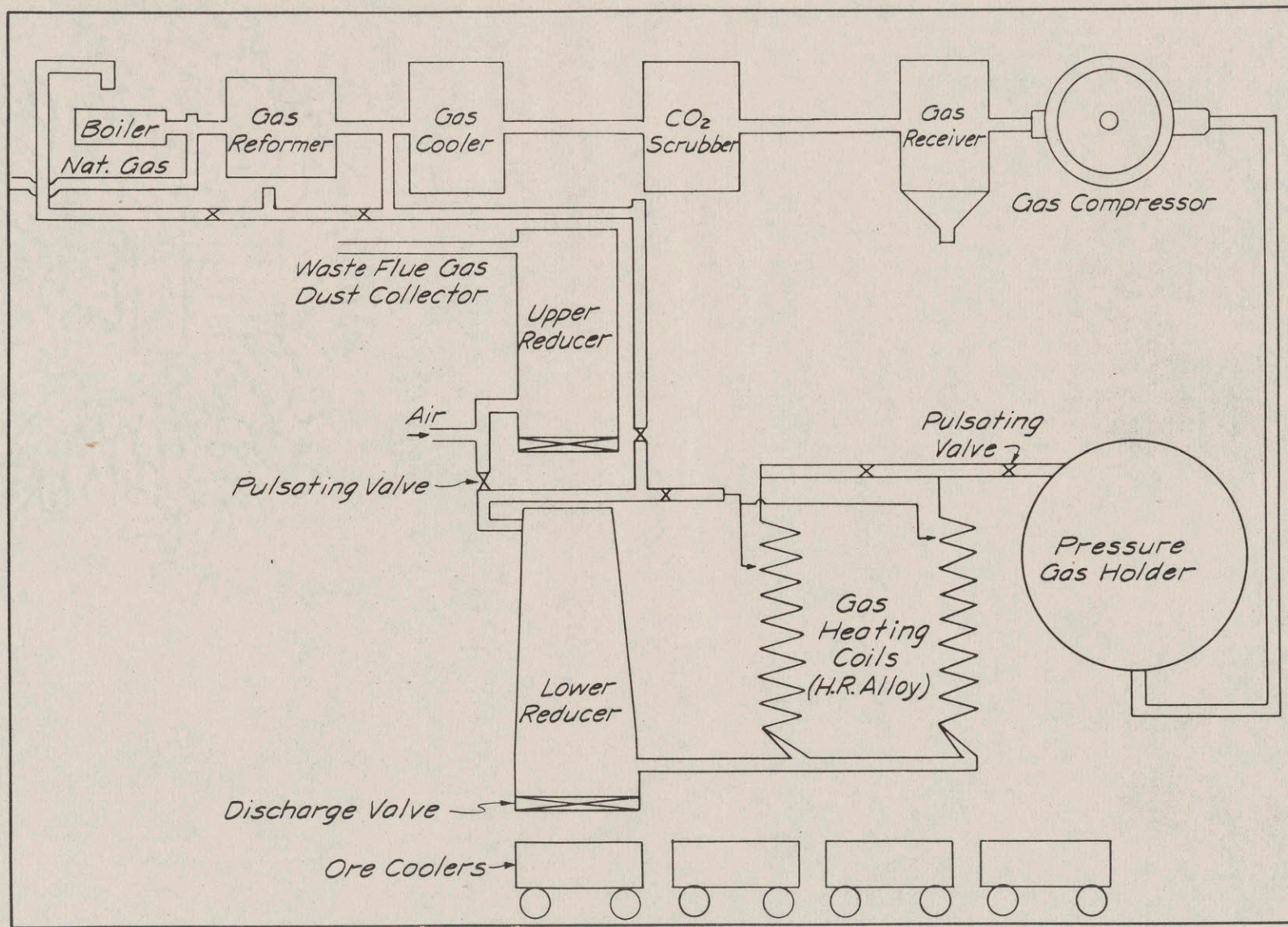


FIGURE 25.- Schematic diagram for rebuilt Longview plant.

Estimate of Capital and Operating Costs for Small Commercial Plant
(Capacity, 100 tons a day)

Adequate data for the close estimation of cost of production of sponge iron by a modification of the process described in this report are not available. The following estimates are based upon such data as can be obtained; they are not proposed as definite figures but only as an indication of the order of magnitude of the costs. The investment cost is based upon the use of equipment already available at the Madaras plant as far as this adaptable to the proposed process. For example, crushing and storage equipment is available.

Estimated Gas-Plant Investment Summary

Estimated cost of gas plant to produce approximately 3,000,000 cubic feet of reducing gas a day.

2 generators rated at 90,000 cu. ft. an hr. \$35,000 each.....	\$ 70,000
2 blowers and motor drives, 10,500 feet at 60 inches.....	7,500
Control instruments.....	1,200
2 boilers, 150 h.p. each.....	19,000
Cooling and condensing system.....	10,000
Relief holder, 100,000 cu. ft. capacity.....	25,000
Wash gas piping.....	7,500
Electric power and light.....	10,000
General piping, water, steam and sewers.....	12,000
Carbon removal sump.....	4,000
Water supply for cooling.....	8,000
Housing.....	8,000
Total.....	<u>182,200</u>
Contingencies at 10 percent of above.....	18,220
	200,420
Engineering construction and overhead at 15 percent.....	30,063
	<u>\$230,483</u>

Estimated Operating Cost for Production of 1,000 Cubic Feet of Reducing Gas

Natural gas for re-forming, 275 cu. ft. at \$0.05 a 1,000 cu. ft.	\$ 0.0138
Natural gas for heating re-formers, 300 cu. ft. at \$0.05 a 1,000 cu. ft.0150
Steam, 18 lb. at \$0.20 a 1,000 lb.0036
Power, water, labor, maintenance, and miscellaneous.....	.0700
Total.....	<u>\$.1024</u>

The investment in plant and the operating cost required for removal of water vapor and carbon dioxide must be added to the costs for re-forming gas. Also, if the plant is to be operated as a pressure system, further additional investment is required. These additional costs of a complete plant to produce gas virtually free from water vapor and carbon dioxide are listed below:

Accessory Equipment for Gas Purification

Carbon dioxide removal unit, including erection.....	\$ 75,000
Gas recirculating and cooling equipment.....	8,000
Piping, pumps, and water circulating system.....	7,500
Compressor, including installation.....	35,000
Gas holder.....	15,000
Total equipment and installation costs.....	\$140,500
Engineering and contingencies, at 15 percent.....	21,075
Total.....	\$161,575

Operating Expenses a Ton of Iron, Gas Plant Only

Reducing gas, including recirculation benefits, 30,000 cu. ft. at \$0.1024 a 1,000 cu. ft.	\$ 3.07
Pumping expenses.....	0.25
CO ₂ scrubbing at \$0.03 a 1,000 cu. ft. recirculated.....	0.90
Investment at 15 percent, 365-day year.....	1.61
Total exclusive of reducing plant.....	\$ 5.83

Estimated Reduction-Plant Investment Summary

2 ore-reduction chambers at \$20,000 each.....	\$ 40,000
2 alloy preheating coils at \$5,000 each.....	10,000
Ore handling and charging mechanism.....	20,000
Insulating materials.....	10,000
Ore-cooling system.....	10,000
Valves and pipe fittings.....	15,000
Structural materials.....	60,000
Control instruments and building.....	3,500
Office and laboratory.....	5,000
	\$173,500
Engineering and contingencies at 15 percent.....	26,025
	\$199,525

Operating Expense a Ton of Iron, Reducing Plant Only

Iron ore, 50 percent Fe natural at \$4.50 a ton of iron.....	\$ 9.00
Crushing and sizing.....	.05
Power.....	.05
Maintenance.....	.10
Personnel:	
Superintendent and office engineering.....	.28
3 foremen.....	.20
6 operators.....	.35
6 laborers.....	.20
1 analyst.....	.07
Investment at 15 percent.....	.82
Total.....	\$ 11.12

The final analysis then shows the entire plant investment as:

Gas plant.....	\$230,483
Vapor and CO ₂ removal.....	161,575
Ore-reduction plant.....	<u>199,525</u>
Total.....	\$591,583

Operating costs on a 100-ton-a-day amortized investment basis show that the cost a ton of metallic iron produced would be:

Gas-plant operation.....	\$ 5.83
Ore reduction, including ore,..	<u>11.12</u>
Cost a ton of iron.....	\$ 16.95

Equipment cost is estimated for new material. At present it would be necessary to use reconditioned equipment to obtain any reasonable deliveries. Plant investment may be reduced somewhat by adopting used equipment wherever possible.

ELECTRIC-FURNACE MELTING OF SPONGE IRON

The behavior of the sponge iron in an electric furnace was investigated by melting material of the following quality in a standard 1-1/2 ton acid-lined furnace.

Iron, total.....	75.9
Iron, metallic.....	63.0
Percent reduction.....	83.0
Insolubles.....	14.1
Carbon, free.....	1.53

Attention should be given to the carbon content of the charge. Each charge of 2,000 pounds contained 30.6 pounds of carbon. Other carbon was added in heats 1 and 3.

A log of the heats is shown below, followed by a discussion of the experiments.

	<u>Heat 1</u>	<u>Heat 2</u>	<u>Heat 3</u>	<u>Heat 4</u>
Total power used, kw.-hr.	1320	910	1070	1000
Time, minutes.....	150	90	130	100
Charge, pounds				
Scrap, pounds.....	582	-	-	-
Sponge, pounds.....	2000	2000	2000	2000
Carbon, pounds.....	100	-	5	-
Ore, pounds.....	20	25	95	-
Mill scale, pounds.....	15	-	-	-
Sand, pounds.....	75	-	20	200
Limestone, pounds.....	10	-	-	11
Ferromanganese, pounds..	14	-	-	8
Silicomanganese, pounds.	-	12	25	-
Pouring temperature, °F. .	3100	3000	2930	3050
Metal yield, pounds.....	1528	1429	1433	1387

	<u>Heat 1</u>	<u>Heat 2</u>	<u>Heat 3</u>	<u>Heat 4</u>
Product analysis:				
Carbon, percent.....	1.35	0.79	0.34	0.19
Manganese, percent.....	0.77	0.59	0.98	0.38
Phosphorus, percent.....	0.119	0.156	0.159	0.150
Sulfur, percent.....	0.052	0.018	0.027	0.022
Silicon, percent.....	1.15	0.75	0.68	0.05
Metallic iron recovery, percent.....	80.1	100.0	100.0	100.0
Total iron recovery, percent.....	69.4	91.9	90.1	88.3

Heat 1

Scrap steel and carbon were added to the sponge charge before melting to help prevent any possible damage to the furnace by the action of iron oxides on the acid lining. This proved to be unnecessary as there was not the least indication of abnormal reaction with the lining. The time consumed in making the heat was longer than necessary since excess carbon was worked out with additions of ore and mill scale. In all tests the ore was sacked in 100-pound burlap bags and pushed into the furnace, the temperature of which was approximately 2,000° F. At the end of the charging period, which was ordinarily 5 to 10 minutes, the charge had begun to get sticky. The sponge conducted enough electricity to strike an arc but not enough to start the melt efficiently. An iron bar about 1-1/2-inches in diameter, placed just above the burden against two of the graphite electrodes, started the current satisfactorily. A small molten puddle in the middle of the furnace sufficed to maintain an arc. After a puddle about 15 inches in diameter was obtained, the ore was worked from the sides to the middle and so on until the entire charge was melted. There was much shrinkage during fusion.

All tests were similar to the procedure for melting iron turnings.

Low recovery of total iron and metallic iron was caused by lack of experience in slagging the melt rather than poor metallurgical reactions.

When the entire charge was melted, visual examination of test specimens indicated that more than 2 percent carbon was dissolved in the iron. The carbon content was reduced to 1.35 percent by addition of ore and mill scale.

Heat 2

Since no corrosion of the furnace lining had occurred in the first heat, it was decided to charge the next heat without scrap or carbon other than the 1.53 percent carbon in the sponge. High carbon persisted, and 25 pounds of ore was added to lower it.

The phosphorus content of the resulting metal was higher than in the previous test because it had not been reduced by diluting with low-phosphorus scrap. Practically no phosphorus entered the slag, which is to be expected in normal acid-melting processes.

Heat 3

The sponge became sticky before complete fluidity was reached, and 20 pounds of sand was added to heat 3 to determine if additional silica would facilitate melting. No great improvement was found.

The 95 pounds of iron ore added to this heat eliminated nearly all of the carbon in the charge and 5 pounds of coke was added to bring the carbon content to 0.34 percent. The desired analysis was carbon, 0.35 percent (final, 0.34); manganese, 1.00 percent (final, 0.98); silicon, 0.75 percent (final, 0.68). This test demonstrated the ease of controlling the alloying elements except phosphorus.

Heat 4

In heat 4 an attempt was made to remove a portion of the phosphorus by forming a large volume of slag. For this purpose, 200 pounds of sand and 11 pounds of limestone were added. More limestone would have been desirable, but it was feared that the acid lining of the furnace would be damaged if more were added. The ore was slagged as early as possible after semimolten conditions existed. In this way three slags were drawn as early as possible. The final analysis showed that very little phosphorus had been removed and the small quantity of limestone added was probably more responsible for the difference than the method of slagging.

Summary of Melting Tests

With experience and practice, the total power consumption probably would be 800 kw.-hr. a ton of metal produced.

After 2,000 pounds, or slightly less, of the original charge was melted, additional sponge could be added, thereby increasing the capacity and output a kilowatt-hour.

In these tests the phosphorus was not removed effectively.

Carbon, manganese, silicon, and sulfur presented no operating problem.

The foaming and boiling of the molten metal ordinarily caused by the presence of base-metal oxides were not prevalent. On the whole, the bath was quiet, and the composition and viscosity of the slag were normal.

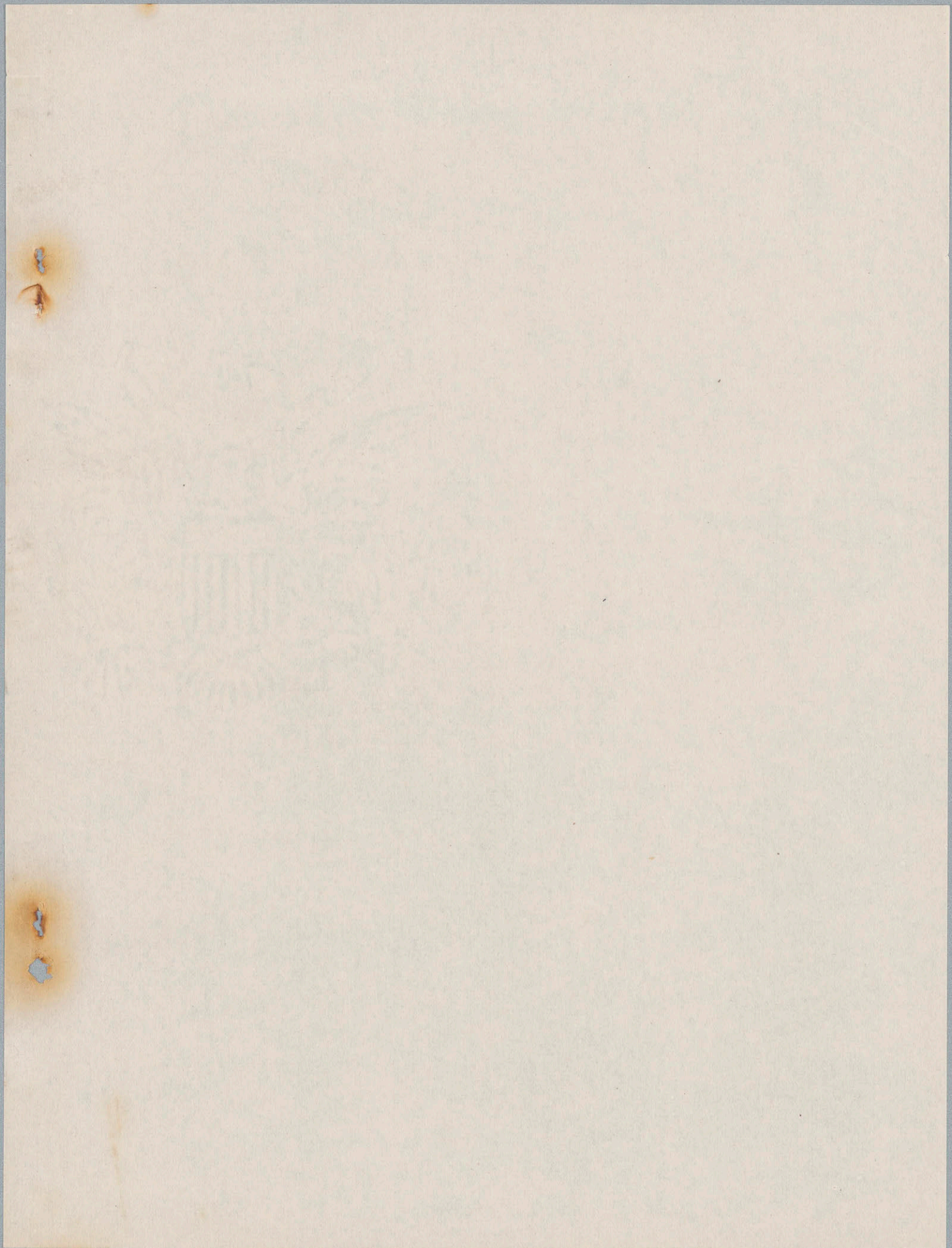
The two greatest difficulties during the melt were the bulkiness of the charge and the stickiness when semimelted.

Castings poured were normal. Pictures of some of the ingots and a casting are shown in figure 26 (fol. p. 36).

Corrosion of furnace lining and ladle was not evident.

CONCLUSIONS

1. A 25,000-pound charge of ore was reduced to about 80 percent in 13 hours.
2. Higher degrees of reduction were not obtained because (a) carbon dioxide and water vapor were contained in the reducing gas in amounts sufficient to retard the rate and extent of reduction and (b) proper temperatures necessary for rapid and efficient reduction of the ore could not be maintained. The first stage of the reduction reaction is strongly endothermic. The heat requirements of this reaction, together with the radiation loss, is responsible for the decrease in temperature at the beginning of each test.
3. Sintered ore did not reduce as rapidly as unsintered ore. Each ore has a slightly different reduction characteristic, therefore, data obtained in this investigation may not hold true for all ores but rather may be an indication of the general trend of reactions under various conditions.
4. The value of pulsating the reducing gas through the ore charge was not determined definitely. The tests made for comparative purposes did not show any distinct advantage.
5. Discharging the partly reduced ore was difficult. Some means of preventing the ore from sticking and a rapid, easy, discharge method would have to be developed in any commercial operation.
6. Sponge iron, about 80 percent reduced, is easily melted in an electric furnace.



A-275

October 13, 1930

Mr. C. V. Drew, Vice-President,
Cerro De Pasco Copper Corp.,
44 Wall Street, New York City.

Dear Mr. Drew:

SPONGE IRON

I record the views expressed to you and Mr. Neave on Thursday to the effect that we should amend the Deppé-Musso agreement to an option to license and to include future inventions by Deppé as well as Musso.

By the present agreement you place the Corporation in the position of withdrawing opposition in Peru by practically confiscating the patent. You for \$200 take an option to purchase good for the life of the patent and forbid the inventor to license anyone else to use it. Incidentally, if Deppé states facts, the United States has already allowed the ^essntial claim and you have no just ground for opposing his Peruvian application if reasonably worded.

There is something fishy about this whole thing. I cannot see why Deppé would consider the agreement you have drawn up and he has not to date produced the U. S. papers. If you make the changes I suggest you will get all you will ever want and be in a much better position should we discover some unexpected motive.

Very truly yours,

Ady

A-311

November 20, 1930

Mr. C. V. Drew, Vice-Pres.,
Cerro de Pasco Copper Corp.,
44 Wall Street, New York City.

Dear Mr. Drew:

SPONGE IRON

I return herewith the November 17th revision of the Deppé agreement. I have no further suggestions to make beyond the possible insertion of "now or hereafter" before "made" on the last line of the first page.

Very truly yours,

Gray

A-297

October 30, 1930

Mr. C. V. Drew, Vice-Pres.,
Cerro dePasco Copper Corp.,
44 Wall Street, New York City.

Dear Mr. Drew:

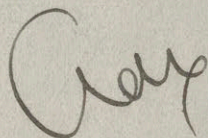
SPONGE IRON

I return herewith the last draft of the Deppé agreement. The equities seem all right. I have only two suggestions.

In line 4 of paragraph "3" it would seem necessary to insert "or improvements thereon" after "Exhibit A".

Some of the tenses in "1" do not strike my ear as in accordance with our meaning although I confess that it is not easy to grasp these two-hundred-word sentences without coming up for air. Where we say "applications for patents have or shall have been filed", do we not mean "have been or shall be filed"? The first construction seems to imply certain time limits.

Very truly yours,



A-162.

51 Maiden Lane.

New York, N. Y., Nov. 13, 1928.

Mr. C. V. Drew,
Vice Pres., Cerro de Pasco Copper Corp.,
44 Wall St., New York, N. Y.

Dear Mr. Drew:

Sponge Iron:

I was much interested in what Mr. Anderson had to show regarding his sponge iron process.

I do not see that you can count on any great amount of waste heat for heating the solution. Apparently the roasting gases are used for preheating the air to the oil burners and the only waste heat is the sensible heat of the product.

Assume a specific heat of 0.17 for the product and a temperature drop of 2000 deg. F. and there will be available only 340 B.T.U. per lb. of sponge iron. As 120,000 B.T.U. are required to heat a ton of liquor from 60 deg. to 100 deg. F., you would require 340 lbs. of sponge iron to heat it.

On the other hand it may be not only a cheap but a chemically active source of iron which may work in cold solutions, and possibly afford a cleaner precipitate.

I recommend that a trial lot of crushed pyrite calcine be sent to Perth Amboy for test, that experiments be

Mr. C. V. Drew, - - Sheet #2. - A-162. - Nov. 13, 1928.

made as to what sort of a flotation concentrate could be made before roasting to see whether the lower furnace costs would pay the value losses. Also that tests be run on the sponge products as to their activity in solutions of various temperatures.

Yours very truly,

Adx/B.

*L. A.
B.*

CONTENTS NOTED
DEC 13 1928
LAWRENCE ADDICKS