#### SUMMARY

This progress report summarizes operating results on the gasification of natural lignite from five runs of the commercial-scale pilot plant at Grand Forks, N. Dak., during December 1946 and calendar years 1947 and 1948, when the plant was operated for 1,996 hours. A primary specific objective was to determine the useful life of the outer metal retort tube as it is influenced by corrosion and other factors under a wide range of operating conditions. A secondary objective was to develop the technology of production when generating gas with a high ratio of hydrogen to carbon monoxide in both the divided and continuous annuli.

Results are summarized in graphs and tables that show hydrogen-carbon monoxide ratios from 2.3 to 9 at gas-production rates from 6 to 12 M cu. ft. per hr. (thousand cubic feet per hour), or approximately 30 to 60 cu. ft. per square foot of heated retort-wall surface per hour. Gas yields ranged from 36 to 54 M cu. ft. per ton of natural lignite or 63 to 91 M cu. ft. per ton of moisture- and ash-free lignite. Production rates were higher in the divided annulus, but these represent the maximum capacity of the plant only at high hydrogen-carbon monoxide ratios when the high concontration of excess steam and low furnace temperature limit gas production. A ratio of 6 was the highest produced in the continuous annulus because mechanical limitations of the auxiliary equipment prevented attainment of higher ratios. The lignite feed rate appears to influence the production rate more than any other individual operating variable. Heat balances for four periods when gas with ratios from 2.3 to 7.2 was produced with a lignite feed rate of about 325 pounds per hour suggest that the potential heat in the gas is lower at higher ratios, owing to increased heat losses in the excess steam. Heat-transfer rates through the retort tube in these four tests were between 1,500 and 3,000 B.t.u. per square foot per hour. These rates are much lower than those reached in many earlier tests, but no attempt was made to achieve maximum rates of heat transfer.

Because the life of the motal retort tube is particularly important in the economy of the process, experience with several retort tubes is discussed. A mild-steel tube protected against corrosion by metcolizing process 45 failed after 449 hours of service, partly at furnace temperatures about 2,000° F. As the coating failed locally and partial collapse was observed in the lower reaction zone, a tube of this kind and size is not suitable for this service. A double-armor 446-alloy Pluramelt tube corroded excessively in 1,781 hours of service, approximately 40 percent of which was at furnace temperatures above 2,000° F. It was reconditioned and used for an additional 1,529 hours at temperatures less than 2,000° F. Further corrosion was slight, but a progressive increase in diameter was noted, probably because of the difference in coefficient of expansion between 446-alloy and mild steel. A final run with a cast EK-alloy tube arranged with a nominal 2-1/4-inch annulus was discontinued after 98 hours of creatic performance because the lignite failed to flow freely through the irregular, rough-walled annulus.

#### INTRODUCTION

Research on the gasification of lignite with steam was begun by the Bureau of Mines in 1943 as one phase of a project to utilize North Dakota Lignite in beneficiating low-grade Minnesota iron ores. It was envisioned that sponge iron could be formed by reducing fine ore with reducing gases rich in hydrogen, which were produced from lignite. Interest in the gasification of lignite has continued because the large reserves suggest that it is an important potential raw material from which synthesis gas and hydrogen for the budding synthetic liquid fuels industry or to manufacture chemicals can be produced.

As earlier attempts to gasify American lignites with steam had met little success, new equipment for continuous gasification was developed. An earlier report (1)2/ describes the development of this process in which the lignite flows by gravity through an annulus between metal walls, where it is gasified in the presence of steam. Heat for the reactions is transferred through the outer wall of the annulus.

Construction of the commercial-scale pilot plant was begun by the Bureau on the campus of the University of North Dakota in the fall of 1944. A preliminary run was made in March 1945, after which the plant was operated 1,861 hours in 1945 and 1946. A description of the plant and operating results during this period have been published (2), and a theoretical analysis of gasification in externally heated retorts has been made (3). At the end of this period the plant was considered ready for systematic tests to develop the technology of gas production and at the same time simulate commercial use of the metal retort tube in extended periods of service.

The commercial-scale plant was operated for an additional 1,996 hours in late 1946, 1947, and 1948. The operating results from this period are the subject of this progress report. In addition, experience with several metal retort tubes that have been used in the plant is reviewed.

#### Acknowledgments

This investigation was made under the general direction of A. C. Fieldner, formerly chief, Fuels and Explosives Division and now chief fuels technologist, Bureau of Mines, Washington, D. C. Helpful guidance and counsel have been rendered by L. C. McCabe, chief, Fuels and Explosives Division, and R. L. Brown, cosl-technology coordinator, Bureau of Mines, Washington, D. C. Dean L. C. Harrington, College of Engineering, University of North Dakota, served as consulting engineer and offered continual encouragement, as well as many suggestions.

Acknowledgment is also made to W. W. McMillan, chemist, North Dakota Research Foundation, Grand Forks, N. Dak., who analyzed the product gas during run 6.

## OBJECTIVES OF TESTS

#### General

The general objective of this investigation is development of a continuous process for gasifying lignite in an externally heated annular retort with metal walls. One phase of this objective was attained with construction and successful operation of the commercial-scale pilot plant at Grand Forks. Since then the objective has been to establish the economy and technology of the process.

Underlined numbers in parentheses refer to references given under Literature cited.

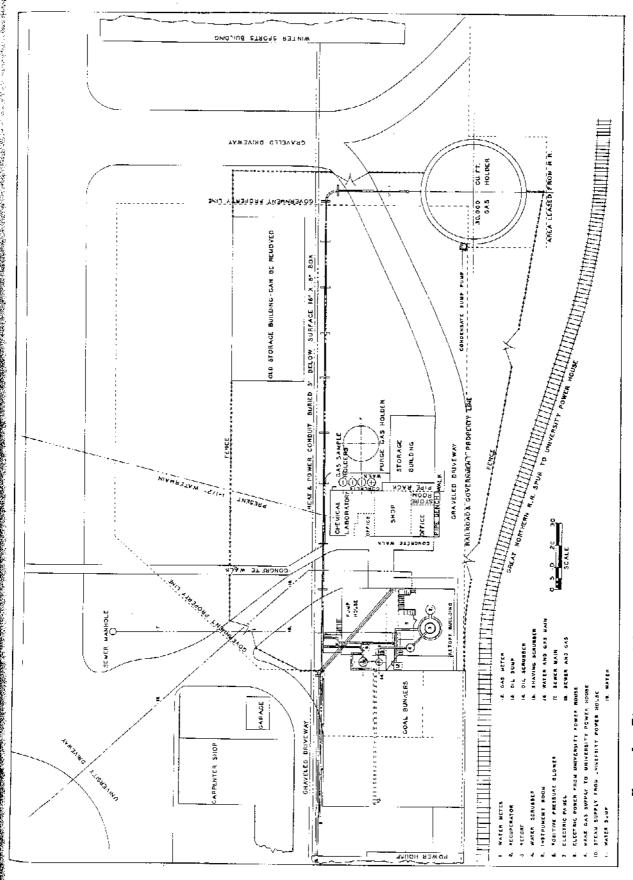


Figure 1. - Plan view of Government property and commercial-scale pilat plant, Grand Forks, N. Dak., 1945.

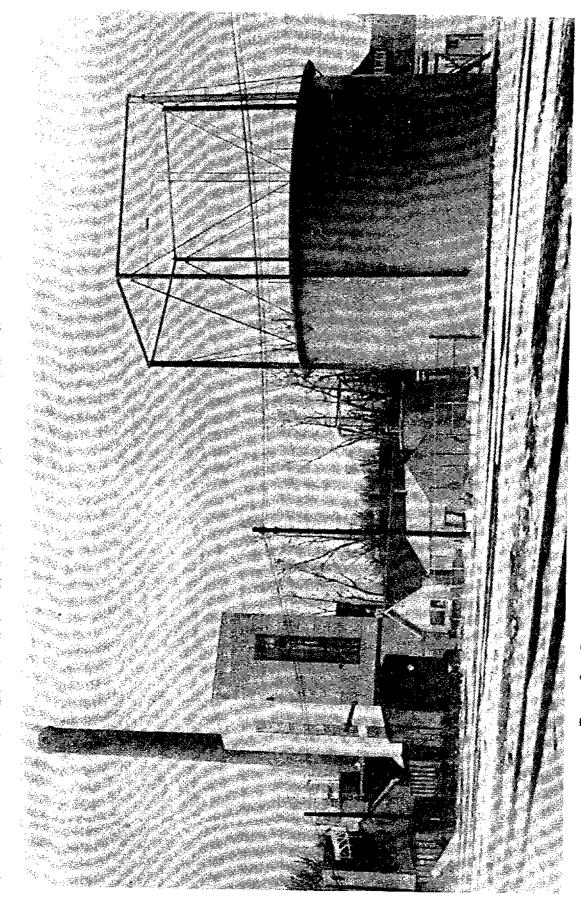


Figure 2. - Commercial-scale pilot plant for gasifying lignite, Grand Forks, N. Dak,

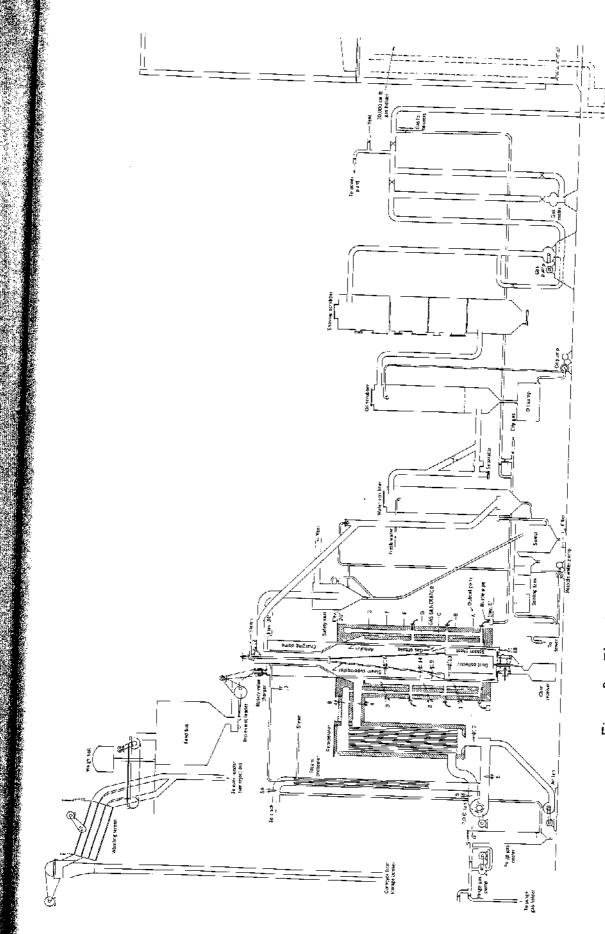


Figure 3. - Flow diagram of commercial-scale pilot plant for gasifying lignite.

### Specific

A primary specific objective of the operations reported herein was to determine the useful life of the outer metal retort tube as it is affected by corrosion and other factors under a wide range of operating conditions. Utilization of a metal tube at high temperature to facilitate the transfer of heat for the gasification reactions is a unique feature of the plant, and it must be demonstrated that metals with satisfactory performance and maintenance characteristics from a commercial viewpoint are available.

Each of the rums had a secondary specific objective related to the technology of the process. In two of the five runs, the secondary objective was to produce gas with a high ratio of hydrogen to carbon monoxide in a divided annulus with upper and lower reaction zones. In two other runs the capacity and flexibility of the plant were investigated when the reaction zone was a continuous annulus. As theoretical considerations suggest that an annulus less than 3 inches wide should produce more gas because the lignite should reach a higher average temperature and the rate of reaction with steam should increase, a fifth run was attempted with a nominal 2-1/4-inch divided annulus.

# DESCRIPTION OF COMMERCIAL-SCALE PILOT PLANT

#### General

The gas generator and auxiliary facilities were constructed on a site provided by the University of North Dakota on the campus at Grand Forks. The site, progress of construction, plant, and other facilities have been described (2). The auxiliary-plant equipment has not been altered in any essential way, although in November 1947 the oil and shaving scrubbers were enclosed to protect them against freezing weather. The plan of the plant and facilities is given in figure 1, and a general view may be seen in figure 2.

# Flow Diagram and Description of Operations

Figure 3 shows the basic flow diagram of the plant, which will be utilized in a brief description of its general operation. The diagram shows the divided-annulus arrangement of the retort unit as used in some tests. Later the arrangement of the retort for each test will be discussed in detail.

Lignite enters the charging dome from the feeder in small increments at a controlled rate and slowly descends into the externally heated annulus. Steam, superheated by waste flue gases, joins the lignite as it enters the annulus from the charging dome. Heat transferred through the outer metal wall of the annulus dries the lignite and then carbonizes it, with formation of gas and tar. As downward flow continues the temperature increases, and reactions between gas, tar, steam, and lignite char form gas with hydrogen, carbon monoxide, and carbon dioxide as the principal constituents. About two-thirds of the length down the annulus, gas and unreacted steam flow through the gas offtake in the inner wall and upward in the center of the retort. The partly gasified char passes on down through the lower annular reaction zone, where further gasification takes place by reaction with steam superheated by heat exchanged with the hot product gas in the center of the retort. The superheated steam flows into a steam chest connected to the bottom of the annulus and thence upward in it. Char, the residue of ungasified lignite, is extracted continuously at the bottom of the unit.

Hot gas leaves the top of the generator and flows through the condenser and scrubbers on its way to the meter. Part of the clean, cool gas heats the generator,

and the remainder is stored in the holder or piped to the University power plant. The heating gas is pumped to a bustle pipe at the bottom of the generator and from there flows to the burners through individual feed pipes. Air for combustion, premixed with approximately four times its volume of products of combustion, is preheated in the recuperator and enters the space at the top of the furnace. The air mixture then flows down through individual flues to each burner.

The diagram shows the locations of thermocouples in the generator and wastegas system. They are numbered to correspond with the numbers given with the data on temperature.

The composition and volume of gas, which is produced continuously, as well as the percentage of gasification of the lignite, are controlled by the lignite feed rate, the steam-flow rate, and the temperature in the furnace. After the rate of char removal (approximately corresponding to a predetermined lignite feed rate), furnace temperature, and steam-flow rate have been fixed, the lignite feed rate finally is readjusted to maintain a constant level in the charging dome.

## The Rotort Unit

Sketches in figure 4 show the arrangements of the retort unit in runs 5, 6, 7, 8, and 9. In the first four of these runs the same outer metal tube was used. It is a cylinder 48 inches in internal diameter and 20 feet 6 inches long, with a heated area of approximately 210 square feet measured on the inside. This tube was fabricated from 5/8-inch, double-armor type-446 Pluramelt that consists of a 3/8-inch mild-steel core clad on both sides with a 1/8-inch integral layer of 446 alloy.6/ Before these runs, it had been used for 1,412 hours.

The inner tube for run 5 (see fig. 4) was a mild-steel cylinder with an outside diameter of 42 inches, which formed a nominal 3-inch annulus with the outer tube. About one-third of its length from the bottom is a double gas offtake, through which the gas flows to the center of the unit. A conical, mild steel dust collector is welded inside the lower section, forming the inner wall of the steam chest. The heavier dust that blows through the gas offtake falls into the dust collector and flows out through the center pipe in the bottom. This arrangement with the gas offtake in the inner wall is called a divided annulus. The length of the upper reaction zone is taken as the vertical distance along the inner tube of the annulus from the seam at the top to the upper edge of the gas offtake. The length of the lower reaction zone is the distance from the seam at the lower edge of the gas offtake to the bottom edge of the inner tube.

The inner tube for run 6 was the same as for run 5, except that the dust collector forming the inner wall of the steam chest was cylindrical (see fig. 4). The outlet for blow-over dust in the bottom of the dust collector was closed by a cone plug valve controlled by a hand wheel at the top of the unit. The inner tube from run 5 was modified for run 6.

The inner tube for runs 7 and 8 was a continuous mild-steel cylinder 42 inches in outside diameter (see fig. 4) that formed a nominal 3-inch annulus with the outer tube. All steam for gasification enters with the lignite at the top of the continuous annulus, and the gas and char are removed at the bottom. No special provisions are needed for blow-over dust, as it is extracted with the char. The length of the continuous annulus is the vertical distance along the inner tube from the seam at the top to the bottom edge.

<sup>6/</sup> Chromium, 23 to 27 percent; carbon, 0.35 percent maximum; nitrogen, 0.25 percent maximum.

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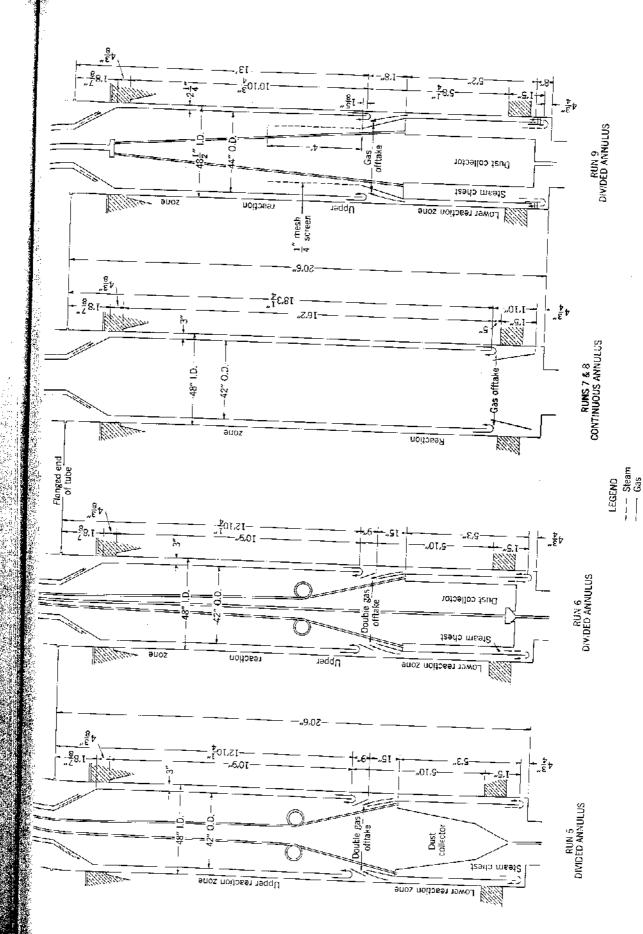


Figure 4. - Arrangements of retort unit.

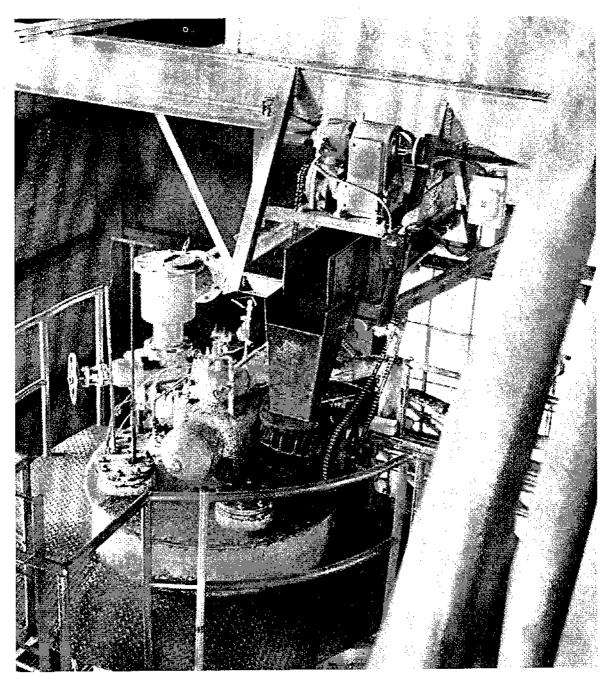


Figure 5. - Reciprocating feeder and rotary-valve charger.

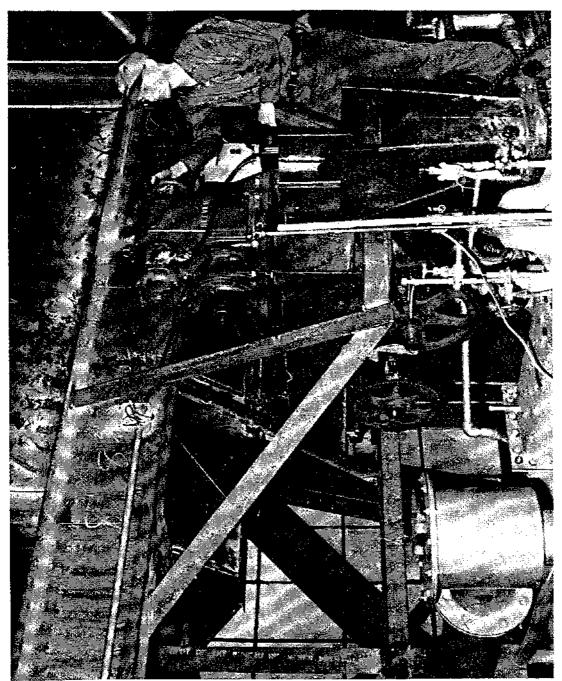


Figure 6. - Drive mechanism for reciprocating feeder and rotary-valve charger.

The outer tube for run 9 (see fig. 4) was the same length as in the other runs, 20 feet 6 inches, but the internal diameter was increased to 48-1/2 inches. It was cast by a centrifugal process from HK alloy, I/ with a wall thickness of 3/4 inch. The slight increase in internal diameter causes a negligible change in the heated area (approximately 210 square feet). The inner tube was 44 inches in outside diameter to form a nominal 2-1/4-inch annulus. With this narrower annulus the inner tube should be hotter at the same furnace temperature; therefore, it was fabricated from  $430 \text{ alloy} \frac{9}{2}/$  to increase its corrosion resistance. The single gas offtake was extended upward with 1/4-inch-mesh screen to catch coarser solid residues and prevent them from blowing over into the dust collector. As in run 6, the steam chest was cylindrical, but the valve at the outlet of the dust collector was omitted. The steam tubes were straight and supported the steam chest and lower part of the inner tube on an adjustable suspension seat mounted at the top of the unit. This design permits limited changes in the area of the gas offtake.

#### Fuel-Handling Equipment

The original coal conveyor did not work well on lignite. Before run 5 a new chain fitted with flat vanes at 10-inch intervals was installed. These changes and addition of a vane feeder at the inlet have resulted in satisfactory performance. The conveyor, screen, and weigh belt indicated in figure 3 were used in these file runs.

#### Fuel-Charging Mechanism

In earlier experiments lignite was fed to the charging dome in batches of approximately 1,500 pounds. Experience with this method suggested that more uniform performance would be achieved if the lignite was fed in small increments at a uniform rate. This was accomplished with a reciprocating increment feeder and rotary block-valve charger, as depicted at the top of the generator in figure 3. Feeder delivery is controlled with a gate and may be adjusted between 2.0 and 3.5 pounds per stroke. The feeder and charger have a common, adjustable-speed drive, so that 200 to 1,600 pounds per hour of lignite can be fed into the charging dome. This dome is purged continuously with about 240 cu. ft. per hour of cold products of combustion (POC). Figure 5 shows the feeder and rotary-valve charger and figure 6, the drive mechanism.

Although the feeding system worked very well in these experiments, the body of the valve, despite greasing at intervals of 3 hours, was abraded by fine lignite dust. As a precautionary measure the valve was replaced before each run.

#### Char Discharge and Blowover-Dust Receiver

Char was removed continuously from the bottom of the unit with the same rotary scraper employed in previous runs. In run 5 the extracted char fell into a hopper, from which it was removed by a pneumatic system, which has been described (2). The blow-over dust flowed from the cone-shaped collector into demountable cans through a 2-inch pipe concentric with the axle of the scraper.

The same system was used to remove the char in run 6, but the collection of blowover dust was modified. This dust was dumped into the char-discharge system periodically by raising the valve in the bottom of the collector. To determine its weight,
the char cone was emptied, the valve lifted, and char-blow-over dust collected together for a measured time. The weight of char was known from previous measurements,
and the blow-over dust was calculated by difference.

<sup>[/</sup> Nominally 25 percent chromium and 20 percent nickel with small amounts of carbon, manganese, and silicon.

<sup>8</sup>/ Chromium, 14 to 18 percent; carbon, 0.12 percent maximum.

With the single annulus in runs 7 and 8, only char is collected at the bottom of the retort. During periods A and B of run 7, the pneumatic discharge system blocked frequently and was abandoned thereafter. It was replaced with a flanged-mouth can botted directly to the discharge cone. The can is removed and weighed periodically to determine the weight of char.

Similar cans were used as receivers for both the char and blow-over dust in run 9. As in run 5, the blow-over dust was withdrawn through a 2-inch pipe concentric with the axle of the scraper. Figure 7 is a photograph of the receivers with that for char in the foreground.

The can used as the char receiver was purged with about 165 cu. ft. per hour of products of combustion. When an independent dust receiver was used, it was purged with about 35 cu. ft. per hour of products of combustion.

## Recuperator

Inspection of the recuperator after run 5 showed that a large number of the Sicromo 5-S tubes had corroded to destruction in the upper and hotter section, although they were in good condition in the lower and cooler section. The corroded parts were replaced by 18-8 alloy tubes welded to the good lower section of the old tubes, except that the 10 offset tubes on the outer circumference were abandoned. The present recuperator has 258 tubes instead of the original 268. When these repairs were made, the upper tube sheet and the four upper baffles were replaced with new ones fabricated from 18-8 alloy. Since this maintenance, the recuperator has required no other attention.

# Instrumentation and Mothods of Obtaining Data

The pilot plant is well-equipped with instruments to contro! its operation and record essential data. Because the process is continuous, emphasis is placed on determination of rates of flow. Figure 8 shows the instrument panel at the central-control station.

To the left are the manometer tubes indicating pressures throughout the plant. The inclined gages indicate furnace pressure and the flow of gas and air to the combustion space. Above these are the two dial indicators for steam flow to the upper and lower reaction zones. To the right of these is the dial of the recording flowmeter for the volume of product gas. The block terminal for thermocouples appears at the lower right with a temperature recorder above it. A carbon monoxide indicator and alarm appears at the upper right.

Not visible in figure 8 are the indicating and recording meters for carbon dioxide in the finished gas and products of combustion. The latter was installed after run 5. The Ranarex meter for specific gravity of the product gas is not visible.

A belt-type weightometer was used to weigh the lignite. Before each run it was calibrated by collecing the delivery for a measured time and weighing it on platform scales.

After run 6 a Roots-Connersville low-pressure positive-displacement gas meter was installed after the scrubbing system. The record of this meter was accepted as the volume of product gas in runs 7, 8, and 9. The recording orifice meter has been retained primarily for plant control during tests.

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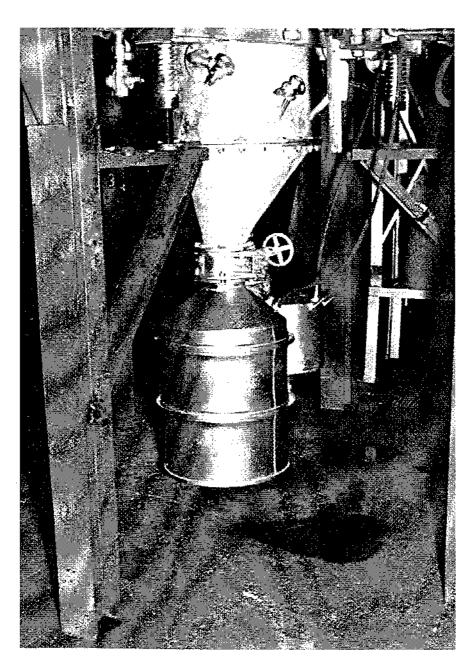


Figure 7. - Char and blow-over dust receivers.

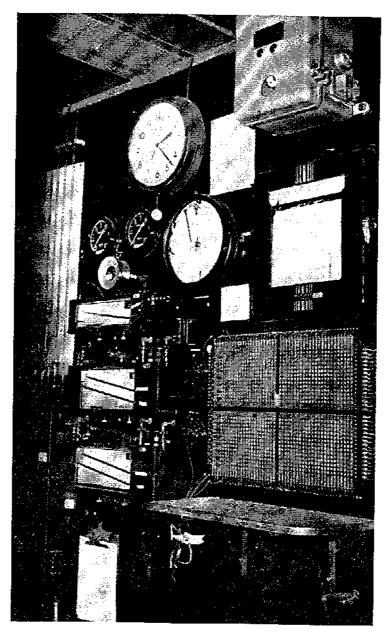


Figure 8. - Instrument panel in control room.

The heating value of the product gas was measured in a Thomas recording calorimeter and checked frequently by calculations from gas analyses made in a Burrell-Orsat build-up analyzer, in which acidulated water saturated with sodium sulfate was the confining liquid. Carbon dioxide, illuminants, and oxygen were determined by the usual absorption methods, using solutions of potassium hydroxide, fuming sulfuric acid, and alkaline pyrogallol, respectively. Carbon monoxide and hydrogen were determined by fractional combustion on copper oxide, whereas methane and ethane were determined by catalytic combustion with oxygen. Nitrogen was determined by difference.

Samples for gas analysis were collected from the outlet pipes of two dry tanks of about 30 cubic feet capacity each. Clean, cold gas flowed through one tank at the rate of 60 cubic feet per 12 hours and through the other at 60 cubic feet per 24 hours. A sample of the effleunt gas from the first tank was taken at the end of each 12 hours during a test period and from the second at the end of each 24 hours. Thus, in a normal test period a minimum of three gas samples was collected and analyzed. The analyses of these three samples, or more if the test period exceeded 24 hours, were averaged to obtain the representative analysis reported for each period. It was found that the results of the three analysis are in good agreement because the test periods were preceded by a stabilization period, were long, and uniform conditions prevailed. As a further precaution, the results were verified frequently by analysis of grab samples taken from the inlet line to the tanks.

Undecomposed steam was determined by weighing the condensate from a measured, cooled volume of product gas. A sample line conducted hot, wet gas from the top of the retort through a water-cooled metal condenser. Condensate from the condenser was withdrawn through the U-trap and weighed. The volume of residual, saturated gas was measured in a wet-test meter, and a correction for water vapor in the residual gas was calculated. From 10 to 50 cu. ft. of gas was used in the determination, depending upon the amount of undecomposed steam in the product gas.

## METHOD OF TEST

To assure reliable results, each test period was preceded by a preliminary stabilizing period of about 24 hours. If steady operating conditions were established, the following period of 24 hours or longer constituted a test period, except for a few instances of less than 24 hours. During a test no operating conditions were changed, except to maintain the preselected lignite and steam rates and furnace temperatures. Changes in conditions between tests usually were limited to one major variable, but in any event no test was begun until operating conditions had been stabilized. All results are averages of periodic readings or of records from recording instruments.

## Accuracy of Results

Extreme care was taken to keep all instruments in good operating condition, and it is believed that the accuracy of the data is within the limits generally accepted as applying to the individual instruments. Orifices for steam have been calibrated, and these results have been corrected. The orifices measuring product gas, heating gas, and air to the furnace and recirculated products of combustion were not calibrated, as no facilities were available. Because the same orifices have been used at all times, volumes thus measured should be consistent and comparable.

Beginning with run 7, the gas volume indicated by the positive-displacement meter was accepted because the meter has a guaranteed accuracy of 0.5 percent. Comparison of the metered volumes with those calculated from the orifice readings showed discrepancies. Differences were not related to the volume of gas or to the conditions of operation but became greater during a run. Discrepancy never exceeded 7.5 percent, and the displacement meter showed consistently higher volumes.

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