Ammonia yields could not be correlated closely with geological age, moisture content, or nitrogen content, although general trends were noted.

Expanding Properties of Coal During Coking

The sole-heated oven, in which the expansion or contraction of a coal under constant pressure is measured, was used to test 29 coals and 29 blends. The coals tested this year were from the following sources: West Virginia, 9; British Columbia, 9; Kentucky, 4; Pennsylvania, 4; Illinois, 1; Washington, 1; and Alaska, 1.

The coal and blend numbers, dry, mineral-matter-free, fixed carbon content, and expansions expressed on a bulk density of 55.5 pounds per cubic foot and as dry, solid-coal expansion are given in table 9. Expansion on a dry, solid-coal basis is preferred in comparing expansions of two coals or in making correlations of the expanding properties of coal with other coal variables, because it eliminates the differences in expansion caused by varying moisture content and specific gravity of the coal.

Samples of Pittsburgh-bed coal from the Warden mine (p28, q28, and r28) were used for blending with low-volatile coals. The variation in the reported results are supposedly due to the time of storage at the Bureau of Mines before the expansion tests were made. The longer the storage period, the less the contraction. Sample p28 was not tested until 3 months after receipt by the Bureau of Mines.

Pocahontas No. 3, Carswell-mine coal (f75 and g75), is the standard blending coal for use with high-volatile coals. The expansion of Pocahontas No. 3 coal has been observed to increase with storage in sealed containers. Sample f75 was not tested until 5 months after its receipt by the Bureau of Mines. Sample g75 was tested every month, and the increase in its expansion with storage is given in detail in table 10.

TABLE 9. - Expanding properties of coals in the sole-heated oven

Coal No.	Expansion, percent: At 55.5 pounds per cubic foot1/	Expansion, percent: Dry, solid coal
p28	- 6.6	40.0
q28	-17.4	23.9
r28	-19.2	26.4
f75	+31.9	104.3
g75	+21.5	88.3
f75A	+ 1.0	52.6
f75B	+ 4.3	57.3
a368	- 8.2	39.9
a368A	+ .9	55.2
a368B	+ 3.8	57.7
378	+ 5.3	54.1
378A	- 6.0	41.7
378B	- 3.9	45.3
379	+20.7	78.6
379A	- .3	48.8
379B	+ 4.8	54.4
380 2/	+ 3.7	50.4
380 3/	+ 8.5	59.8
380 2/	+ 3.2	49.0

See footnotes at end of table.

TABLE 9. - Expanding properties of coals in the sole-heated oven - Continued

Coal No.	Expansion, percent: At 55.5 pounds per cubic foot[1]	Expansion, percent: Dry, solid coal
380A[3]	+ 9.4	60.0
380B[3]	+ 8.3	59.3
387[2]	+ .7	43.7
387[3]	+ 6.0	52.6
381	-10.0	39.1
381A	- 4.0	49.5
381B	- 2.6	50.9
382	- 5.6	43.5
382A	- .8	51.2
382B	+ 1.3	55.2
383	- 7.3	41.9
383A	- .8	51.1
383B	- 1.4	53.9
384	- 6.1	41.3
384A	+ 1.3	53.5
384B	+ 4.3	57.7
386	+39.6	121.7
388	-17.2	31.5
389	-14.3	39.2
392	-19.3	37.4
393	- 8.3	49.5
394	- 7.9	49.3
395	+ 3.7	54.9
398	-13.4	35.6
399	- 5.3	46.1
401	- 6.6	53.9
402	+ 4.6	59.3
413	+11.9	69.6
413A	-16.4	26.4
413B	-13.9	29.2
413C	- 6.0	43.7
415	-19.1	45.1
425	+24.9	89.0
425A	-11.2	38.4
425B	- 3.5	45.4
426	-11.7	33.0
426A	- 1.6	46.8
427	- 7.0	42.8
427A	- .9	53.1
428	- 1.5	47.2
428A	- 8.9	40.9
429	- 4.7	45.9
429A	- 7.2	42.2
429B	-11.0	34.5
429C	- 8.9	41.1

1/ End-of-test contraction for contracting coals; maximum expansion for expanding coals.
2/ Tested as received. Maximum-size particles about 1 inch by 1 inch.
3/ Coal crushed in hammermill.

TABLE 10. - Effect of storage on expansion of Pocahontas No. 3 coal (g75)

Days in storage	Number of tests in average	Average maximum expansion at 55.5 pounds per cubic foot, percent	Increase in expansion from original	Differ significantly; 95-percent range
0	2	21.5	-	Standard
64	2	26.5	5.0	Yes
101	2	24.0	2.5	Yes
128	2	27.6	6.1	Yes
172	2	29.6	8.1	Yes

Statistical analysis shows that differences between average expansions at 55.5 pounds per cubic foot on the same coal will be 1.5 percent or less 95 percent of the time. If this criterion is applied to the differences between averages of two tests on Pocahontas No. 3 coal (g75), the increases in expansion are seen to be significantly different from the standard in each case, indicating that storage of Pocahontas No. 3 coal changes the expanding properties significantly.

A sample of Illinois No. 6 coal (415) was studied in connection with the investigation of the apparent effect of carbon tetrachloride on expansion. This investigation was started because a Pocahontas No. 3 coal, which had been floated on CCl_4 at 1.50 specific gravity, expanded an unusually low amount for its rank. One portion of the Illinois No. 6 coal was cleaned by the heavy media process (magnetite-ore suspension) at 1.55 specific gravity, and another portion was floated on carbon tetrachloride at 1.55 specific gravity. Samples of the raw coal were tested immediately and after storage of several months. Zinc-chloride solution was added to the coal to study the effect of a chloride on expansion. Applying the results of statistical analysis of over 400 test results and based on the 95 percent range, the following conclusions are justified:

1. When the coal is dipped into carbon tetrachloride and thoroughly air-dried before testing, the expanding property is not changed significantly.

2. The presence of carbon tetrachloride as a liquid on the coal when charged lowers the expanding property significantly.

3. Flotation of Illinois No. 6 coal on carbon tetrachloride at 1.55 specific gravity increases the expansion more than does flotation on heavy media of 1.55 specific gravity.

4. The presence of zinc chloride (one pound to approximately 50 pounds of coal) does not lower the expanding property significantly.

Six blends of coke breeze and pitch, four blends of coke breeze and coal, and four blends of coke breeze, pitch, and coal were carbonized in the sole-heated oven, and tumbler tests on the resulting product were made in cooperation with a company interested in agglomerating their coke breeze with pitch or a mixture of pitch and coal. The agglomerated coke breeze could then be used in their carbide furnaces.

The higher tumbler indexes of the agglomerated coke breeze approach those of the lower range of coking-coal blends, but do not compare favorably with the tumbler indexes of the original beehive coke from which the agglomerates were made.

Plasticity of Coals

Plastic properties of 45 coals and 33 blends were determined during the fiscal year. All samples were tested by either the Gieseler and/or Davis plastometer methods; two or more tests on the same sample were made by each test method. A total of 318 tests - 168 by the Gieseler and 150 by the Davis method - was made.

The plastic properties of coals and blends may be conveniently discussed according to characteristic temperature indications of certain stages in the fusion range, the degree of maximum fluidity (dial divisions per minute) developed in the Gieseler test method, and the maximum resistance (pound-inches) observed in the Davis test method. In general, as the rank of the coal increases from high-volatile A to low-volatile bituminous, the fusion range is displaced to higher temperatures, the maximum fluidity becomes less, and the maximum resistance becomes greater. These changes in plastic properties usually correlate well, with corresponding increases in strengths of the cokes produced at 800° and 900° C. from coals of the respective ranks. Variations in chemical and petrographic compositions among individual coals of a given rank have a greater influence on the maximum fluidity and maximum-resistance values than on the temperature range of fusion.

The three coals, a366, 380, and 387, and six blends, f75A, f75B, a368A, a368B, 380A, and 380B, all show good fusion by both test methods and should produce strong cokes. The two samples, 368x and 368y, prepared for testing from Pittsburgh coal (368) by partial carbonization, show complete loss of fusion properties, that is, no resistance in the Davis plastometer test. Samples 368x and 368y should be suitable and samples a368, b368, and 397 unsuitable for gasification by the Lurgi process.

The effect of certain materials on coal-plasticity indexes was studied. Blend 429B, containing 30 percent Sewell coal 429 and 70 percent Pittsburgh coal 28, shows a maximum fluidity of 9,280 dial divisions per minute. As expected, the addition of 11 percent tar increases the maximum fluidity to 12,000, and the addition of 9 percent carbon tetrachloride reduces it to 2,500 dial divisions per minute. This large amount of carbon tetrachloride can conceivably extract some material from the coal or otherwise change its properties in such manner that its plastic properties also are changed.

Coke Properties

Properties and typical analyses of various types of coke were summarized in a recent publication. /2/

Oxidizing Properties of Bituminous Coals

The oxidizing properties of 13 coal samples were determined during the year. Twelve samples, after first drying in nitrogen gas at 100° C. in a closed rotary-drum unit, were tested for their spontaneous heating tendencies in oxygen gas in an adiabatic calorimeter. The characteristic rate of oxidation in air at 100° C. of one coal (406) was determined in a steam-jacketed, rotary-drum unit.

Table 11 summarizes the self-heating rates at 70° and 100° C. of the 12 samples as determined in the adiabatic calorimeter and the ratios of these rates to those of

/2/ Brewer, R. E., Solid Fuels   11. Coke: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-37 to 2-39, John Wiley & Sons, Inc., New York, N. Y.

4369

fresh Pittsburgh bed, Warden mine, coal m28, previously tested by the same procedure. Excepting coals 430, 430s, 431, and 431s, which were tested in the sizes given in table 6, all samples were first stage-crushed to 0- to 1/8-inch size. The first five samples listed in the table were tested to determine the effect of prolonged storage on their rates of oxidation. Continued storage of these coals shows that slow oxidation has occurred, as is evidenced by the drop in their self-heating rates. The third test on coal 346 using dry oxygen gas was made to determine the effect of first drying the oxygen completely before admitting it to the coal charge in the calorimeter. Lower self-heating rates are obtained with the dry oxygen. The normal test procedure is to measure the oxygen at room temperature with a wet-test meter before passage to the calorimeter, so that some moisture is always retained in the oxygen at temperatures below 100° C. Pittsburgh coal p28, if fresh, should give the same self-heating rates as the Pittsburgh coal m28 used as the standard of comparison in the table. As would be expected from its rank and long period of storage, the low-volatile Davy Sewell coal 345 oxidizes very slowly. The low-ranking, high-volatile A fresh coal 398 oxidizes very rapidly, as compared with the standard high-volatile A coal m28 which is quite resistent to oxidation. The high-volatile B coal 414, both before and after the drier, shows very rapid rates of self-heating. The effect of finer-size coal in increasing the rate of self-heating is shown by comparing coal 430s with coal 430 and coal 431s with coal 431. The very low rates of self-heating of coals 431 and 431s are due to their high ash content - 63.1 and 63.0 percents, respectively, on the dry-coal basis - as compared with coals 430 and 430s, which contain only 11.5 and 14.1 percent of ash, respectively, on this basis.

TABLE 11. - Self-heating tendencies of coals to heat spontaneously

Coal No.	Condition of coal 1/	Self-heating rate at - 70° C., °C./Hr.	Self-heating rate at - 100° C., °C./Hr.	Ratio of self-heating rate to fresh Pittsburgh bed, Warden mine, coal at - 70° C.	Ratio ... at - 100° C.
346	In storage 13 months	0.50	2.67	0.72	0.97
346	In storage 19 months	.46	2.24	.67	.81
346	In storage 19 months (tested with dry oxygen)	.29	1.10	.42	.40
p28	In storage 6 months	.46	2.70	.67	.98
345	In storage 19 months	.106	1.41	.154	.51
398	Fresh coal	2.79	13.32	4.04	4.83
414	Raw wet feed to driers	5.20	26.0	7.53	9.42
414	Flash dried, 5 minutes at 550° F.	6.00	25.2	8.70	9.13
430	Feed to washery, 0- to 3/8-inch size	1.30	4.86	1.88	1.76
430s	0- to 1/8-inch portion of coal 430	1.58	6.21	2.29	2.25
431	Free discharge refuse, 0- to 3/8-inch	.23	2.70	.33	.98
431s	0- to 1/8-inch portion of coal 431	.29	2.56	.42	.93

1/ The coal sample in all instances was first dried in nitrogen at 100° C. in a closed rotary-drum unit before charging in the adiabatic calorimeter.

The physical properties of 900° C. cokes made by the Bureau of Mines-American Gas Association method from 12 high-volatile A Appalachian coals and their blends with 20 and 30 percent Pocahontas No. 3 coal were studied.73/ The following conclusions were drawn from a study of these data: (1) Increases in the coking power

73/ Reynolds, D. A., Coal Carbonization: Effects of Blending Pocahontas No. 3 Coal with 12 High-Volatile A Coals: Bureau of Mines Rept. of Investigations 4552, 1949, 8 pp.; Proc. Am. Gas Assoc., 1949, pp. 725-729.

of high-volatile A coals effected by blending with 20 percent Pocahontas No. 3 coal generally were greatest for coals that yielded the weakest coke when carbonized singly; (2) the 1-1/2-inch shatter, 1-inch tumbler, and 1/4-inch tumbler indexes of the cokes from blends of a significant number of the high-volatile coals increased only slightly on raising the proportion of Pocahontas No. 3 from 20 to 30 percent; (3) the 1-1/2-inch shatter indexes of cokes from the high-volatile coals and their blends could be correlated approximately with the 1-inch tumbler indexes, but neither of these indexes was related to the 1/4-inch tumbler index.

GASIFICATION OF COAL, COKE, AND LIGNITE

Two chapters covering gaseous fuels and gas producers were prepared for the twelfth edition of Kent's Mechanical Engineers' Handbook. These chapters include data on the physical and chemical properties of gaseous fuels and provide brief descriptions and operating results of typical gas-manufacturing equipment.74/75/

Lignite

During the year the Lignite Pilot Plant at Grand Forks, N. Dak., continued the investigation of the continuous gasification of lignite in an externally heated annular retort. Two test runs were made with the 310 alloy retort tube installed the previous year and tested in only one run that year. In test No. 12, approximately 85 tons of dried lignite as gasified to give nearly 5.8 million cubic feet of gas in 438 hours of operation. Test No. 13 had a duration of 730 hours during which time over 164 tons of raw lignite as treated to give nearly 7.0 million cubic feet of gas. Though these tests represent an increase of 38 percent in operating time over the previous year and an increase of 61 percent in gas made, there was an increase of only 31 percent in the lignite used. The 310 alloy retort tube appeared to be in entirely satisfactory condition at the end of these test operations.

The marked increase in gas production, with only a moderate increase in hours operated and lignite consumed, is the result of the use of dried lignite in run 12. The use of lignite for gasification has the following advantages over natural lignite: Higher capacity in terms of cubic feet of gas per day and, therefore, reduction in fixed charges per cubic foot of gas made; lower ratio of H_2/CO and, therefore, lower operating costs; more uniform temperature throughout the annular space, and, therefore, more uniform operation; and, more efficient operation in general.

Underground Gasification

The underground gasification of coal has long been advocated as an attractive means of bringing the energy stored in underground coal beds to the surface. With increasing interest in synthetic liquid fuels, the possibility of providing synthesis gas for the various processes by this means has received serious consideration. The advantages which may be realized through underground gasification are: Utilization of dirty coal beds, or coal remaining in worked-out mines; production of energy in a convenient form; reduction of capital expenditure necessary in the mining, transportation, and processing of coal in solid form; and avoidance of the unattractive labor of mining coal.

74/ Newman, L. L., Gaseous Fuels: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-61 to 2-87, John Wiley & Sons, Inc., New York, N. Y.

75/ Newman, L. L., Gas Producers: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-87 to 2-93, John Wiley & Sons, Inc., New York, N. Y.

An underground gasification plant consists of: A means of inlet to and discharge from the underground system; initial underground development to provide passageways through the coal bed; a source of gasification media, such as compressed air, oxygen, or steam; and means for handling and processing the product gas.

Before a commercial plant may be designed, the factors controlling the process must be determined. The length, width, and shape of the underground gasification unit control the location of the inlet and discharge passages. The optimum rate of gasification in the unit determines the volume of blast medium required and the facilities for handling blast and product gases. Operating characteristics, such as pressure drop through the system, heat loss, and gasification efficiency, must be known. Perhaps the most important factor in the design is the development of techniques for controlling gas quality.

The Bureau of Mines and the Alabama Power Co. have jointly conducted and completed one field-scale experiment in the underground gasification of coal and at present are cooperating in a second field-scale experiment. The Bureau of Mines has, in addition, carried out laboratory-scale investigations relating to the same subject.76/77/

Second Field-Scale Experiment at Gorgas, Ala.

The results of the first field-scale experiment and the laboratory work that followed led to construction of the present larger-scale underground gasification project at Gorgas, Ala. This experiment is being conducted in the Pratt coal bed in an area isolated from the main body of coal by nature and consisting of approximately 100 acres. The coal bed is 42 inches thick and relatively level; it lies under an average cover of 150 feet.

The objectives of this second underground gasification experiment are:

1. To determine the quantity of coal that can be gasified from a given initial combustion zone and the shape and extent of the burned-out areas formed during this gasification.

2. To test the design and installation of types of fixed product-gas outlets, including the seals required.

3. To determine the operational characteristics of the experimental installation under such variation of conditions as the nature of the installation and the progress of the work may indicate to be desirable - for example, the length of passage required, the optimum rate of flow, and the pressure drop.

4. To determine the quality and quantity of the product gas generated and the secondary byproducts evolved under the experimental conditions.

5. To obtain all possible information regarding the action of heat on the overlying strata.

76/ Fies, Milton H., and Elder, J. J., Experiments in the Underground Gasification of Coal: Armed Forces Chem. Jour., vol. III, No. 8, April 1950, pp. 6-9, 30-31.

77/ Synthetic Liquid Fuels. Annual Report of the Secretary of the Interior for 1949. Part I. Oil from Coal: Rept. of Investigations 4651, 1950, pp. 43-49.

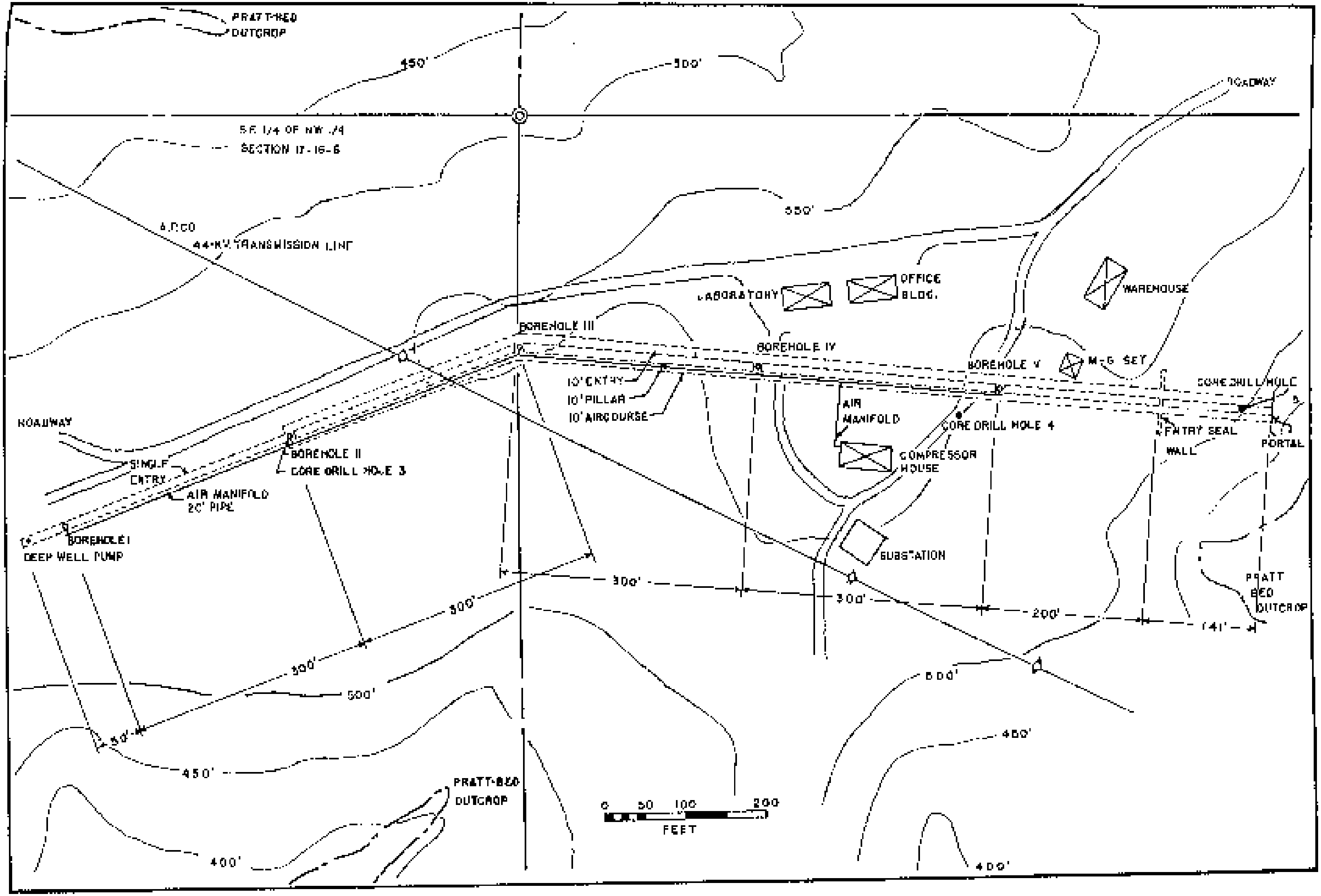

Figure 13. - Site of second experiment in underground gasification of coal, Gorgas, Ala.

6. To develop, without interfering with the foregoing objectives, such fundamental technical and economic information as is likely to be helpful in selecting plant sites, plant installations, and operating processes.

The experimental unit (see Fig. 13) consists of a straight-line passage in the coal, with surface connections at one end through an outcrop seal, and at various points along its length through large-diameter vertical boreholes. Small-diameter vertical boreholes were drilled into the coal bed adjacent to the entry, in order to measure the progress of combustion underground. Air is supplied by a reciprocating compressor, rated at 7,200 std. cu. ft. per min. with a discharge pressure of 30 p.s.i.g. Each of the large-diameter boreholes may be used as an inlet or outlet to the underground system.

Combustion was started in the 300-foot length of coal-bed entry, located at the end of the passage farthest from the outcrop. The air blast was maintained for 10 days in one direction, entering at borehole I, and the product gas was discharged at borehole II. During this period the temperature distribution realized and an increased percentage of oxygen in the product gas led to use of periodically reversed blast operation. By this means heat was stored underground and the temperature distribution improved. After several months' operation the oxygen content of the product gas again increased. To remedy this, dry sand was fluidized by means of compressed air and injected into the entry through 6-inch vertical boreholes drilled for that purpose. A total of 156 tons of sand was injected between boreholes I and II, and the operation was materially improved thereby.

In October, November, and the early part of December, the length of time between reversals of flow was increased, and it was found that a combustible gas was being generated on the coal faces and subsequently burned by bypassing oxygen underground. During this period the 20-inch outlet stacks were operated at temperatures of 1,600° to 2,400° F., with gas burning in and above the stack. Coal consumption near the base of the outlet stack increased, and the gaseous products were evolved at temperatures high enough to make possible their use in raising steam or in operating a gas turbine. A large percentage of the heat of combustion of the coal was brought above ground in the effluent gases.

Between boreholes I and II, from March 1949 through March 1950, 5,125 tons of coal were burned. Operating data and the information obtained from the test holes indicate the coal faces receded by 40 feet midway between boreholes I and II and by 65 feet near the boreholes themselves. This means that a total area approximately 130 feet wide at the boreholes, 80 feet wide at the midpoint of the passage, and more than 400 feet long has been burned out, and to date no limit as to the quantity of coal which can be utilized from a given initial passage has been reached. Furthermore, no difficulty has been met in maintaining combustion of the coal underground, and except for minor maintenance shutdowns operation has been continuous on a 3-shift-per-day basis.

From March 1949 to June 1950 the entire operation of the experimental installations was conducted between boreholes I and II. From time to time additional 6-inch churn-drill holes were put down, either along the line of the original entry or in regions where the coal has been burned out. These holes indicated that the overlying strata has been subjected to temperatures high enough to discolor and modify the physical characteristics of the strata, at least, for 15 feet. Where they were drilled along the line of the original entry it was found that roof action in this region had been such that a condition favorable to bypassing the coal faces had been set up. The test holes drilled in regions other than along the original entry and in regions where the coal had been burned out indicated that roof action had been much more favorable to the process.

Experimentation is being carried on to determine the effect of a shorter length of combustion zone, by means of the construction of large boreholes off the line of the original entry at appropriate locations. One such hole was completed and tested, and using a combustion face 150 feet in length, a combustible gas was produced for 8 hours. Other experimental work in progress is directed toward the concentration of the combustion zone in a specific location and also to control bypassing of air by plugging underground crevices through the use of fluidized, finely divided solid media. The results so far obtained through this type of experimentation indicate that much better operating conditions can be attained than were realized in the original operation between boreholes I and II.

RESEARCH ON THE PRODUCTION OF SYNTHESIS GAS

Gasification of Pulverized Coal

Pulverized-Fuel-Gasification Pilot Plant

During the year the atmospheric-pressure pilot plant was operated for over 30 test runs.

These runs demonstrated that steam could be preheated to 3,600° F. in the pebble stoves and that, using this steam, the oxygen requirements for gasification of the coal were the lowest for any continuous process reported to date. To secure adequate data for process evaluation, test runs were made using steam at temperatures of 240°, 2,000°, and 3,000° F. As will be noted from table 12, the percent carbon gasified was very good.

A total of 56,000 pounds of coal was gasified at feed rates of 200 to 450 pounds of coal per hour.

TABLE 12. - Averaged requirements per 1,000 std. cu. ft. of ($CO+H_2$) produced

Steam temperature, °F.	Coal, pounds	Oxygen, std. cu. ft.	Steam, pounds	Carbon gasified, percent
2,904	33.3	171	81.1	82.5
1,899	37.3	326	42.5	90.7
235	42.3	394	29.0	83.4

One of the outstanding features of this process is its flexibility as regards the ratio of hydrogen to carbon monoxide, which can be produced by control of the reactant ratios and temperatures. This is shown in figure 14. This feature is of value since it makes the process applicable to a wide range of synthetic operations.

Studies have been made of the variation of the coal-air ratio as the mixture flows through the coal-delivery tube. To measure this ratio, an instrument[78] has been developed which utilizes the change in capacity of a condenser when a mixture of coal and air flows between the plates. The instrument uses two synchronized oscillating circuits, which are coupled inductively. When the two oscillating circuits are in phase, no current flows through the coupling circuit. When the frequency of one circuit is changed by changing the capacity of a condenser in the circuit, current flows in the coupling circuit giving a measure of the change in capacity of the condenser. This current is recorded as a spot on an oscilloscope above a fixed datum, and the spot is moved across the field of the oscilloscope in 1 second.

78/ Dotson, J. M., Holden, J. H., Seibert, C. B., Simone, H. P., and Schmidt, L. D., New Method Measures the Solid:Gas Ratio in High-Solid Flow: Chem. Eng., vol. 56, October 1949, pp. 128-130.

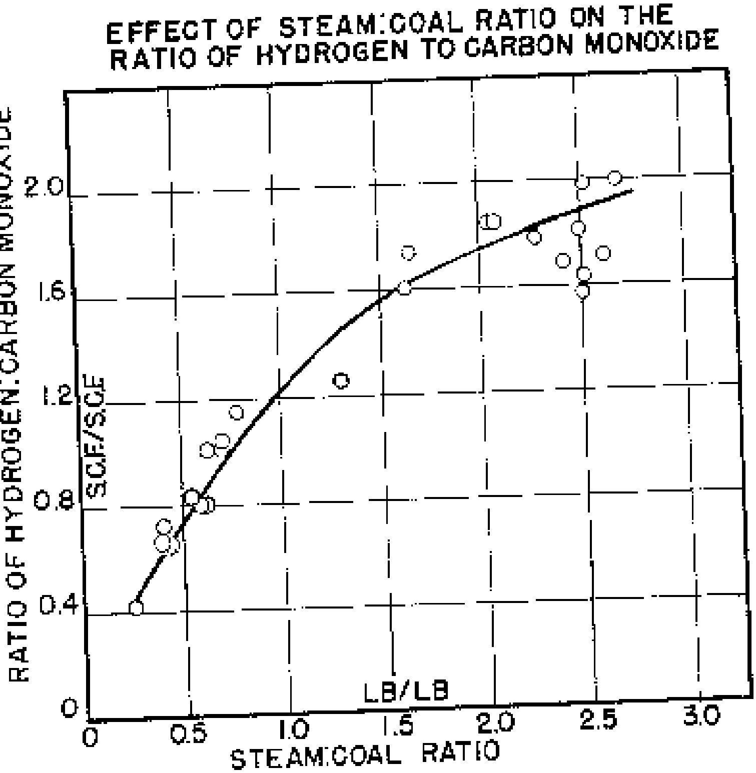

Figure 14. - Effect of steam:coal ratio on the ratio of hydrogen to carbon monoxide.

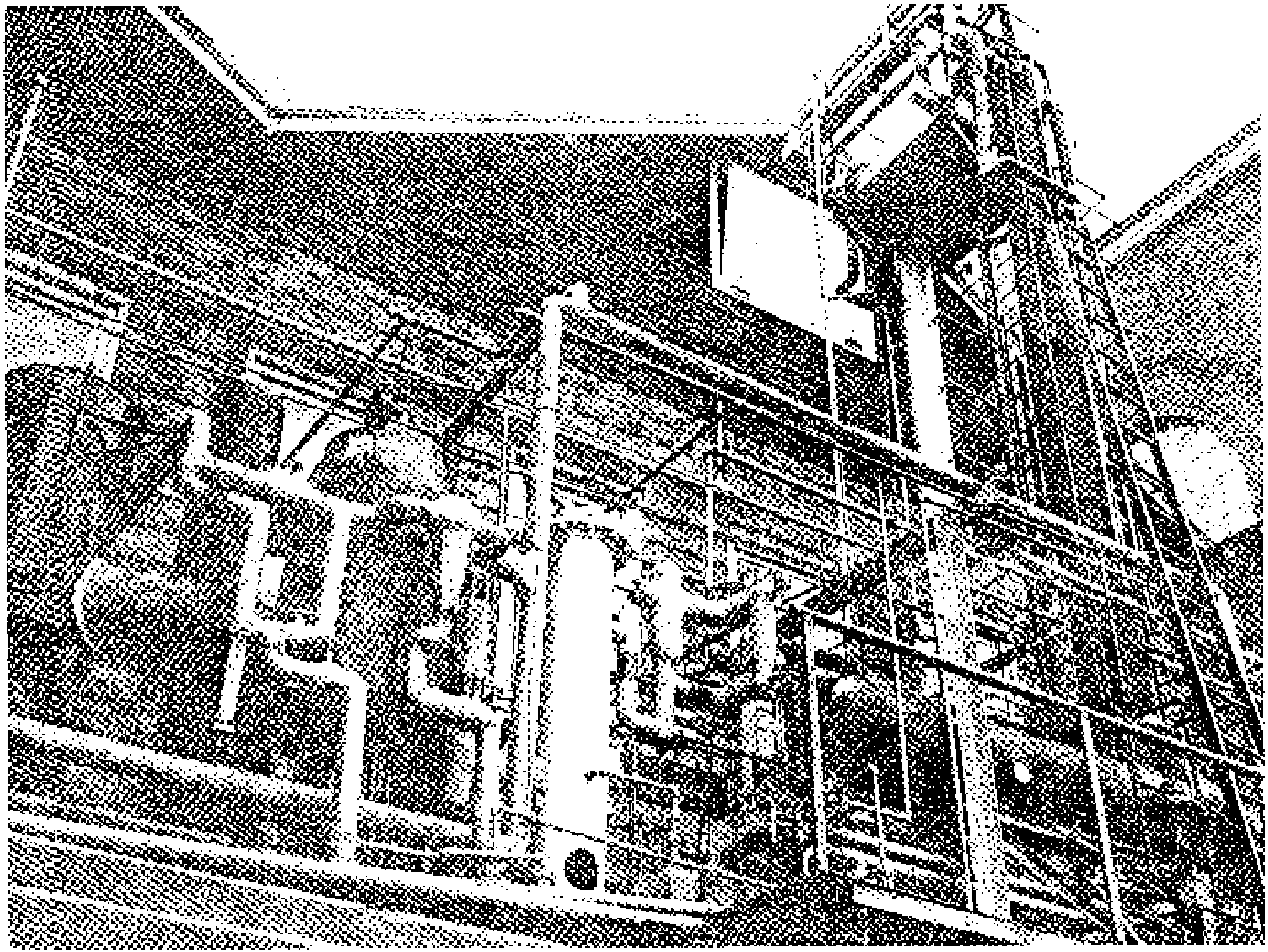

Figure 15. - Gas-purification pilot plant.

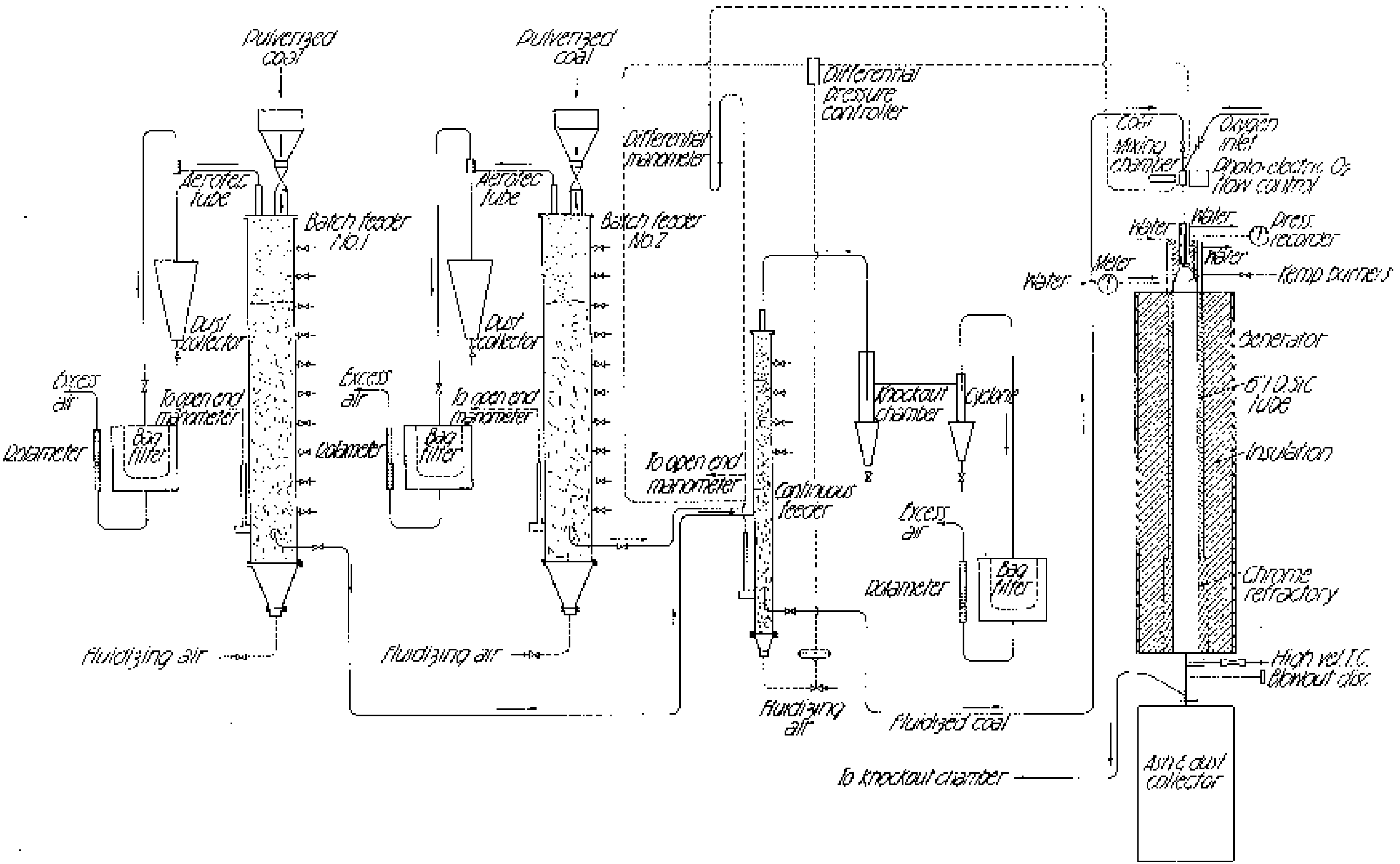

Figure 16. - Flow diagram of pneumatic feeding system and generator for powdered-fuel gasification in laboratory-scale pilot unit.

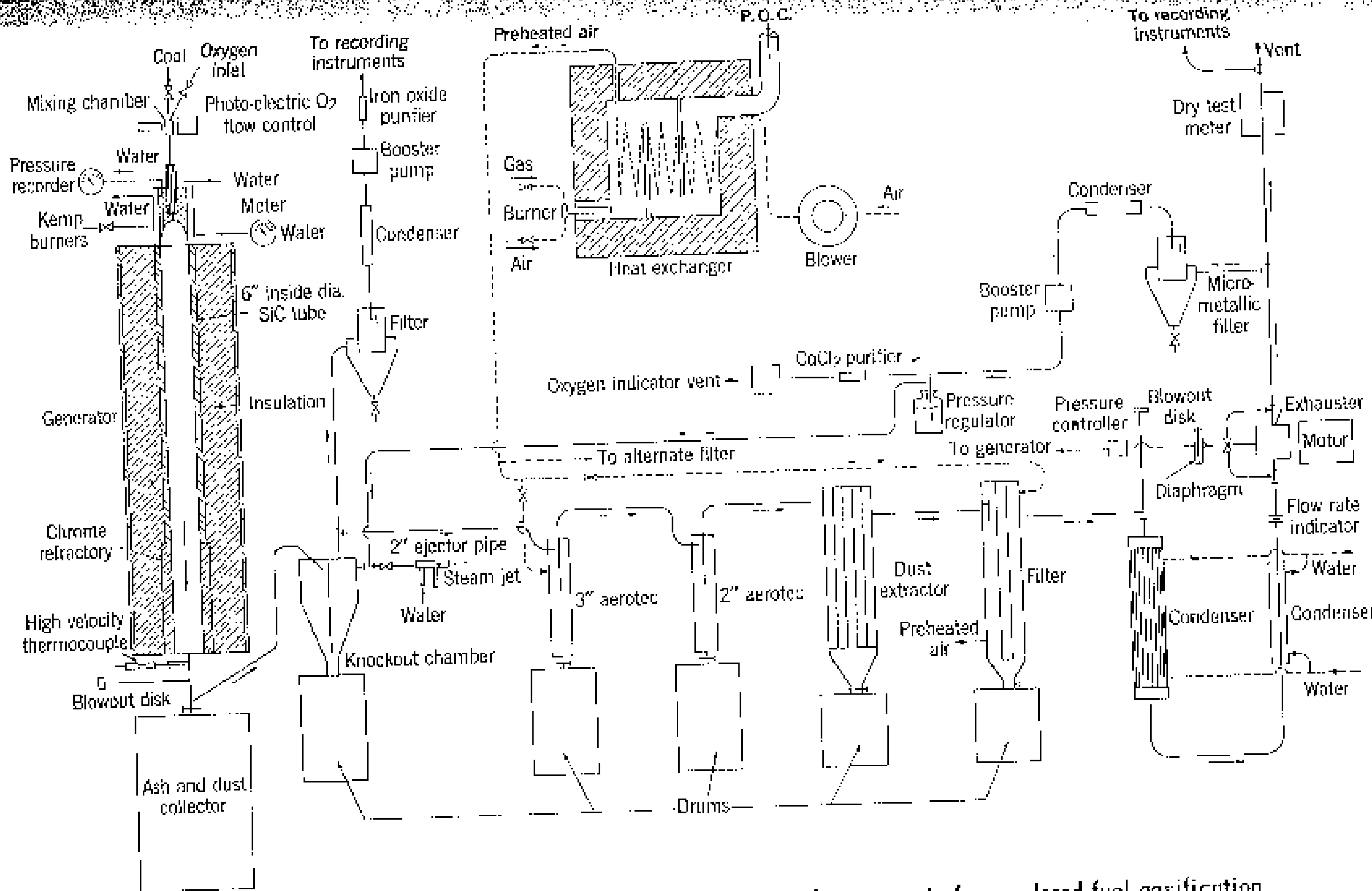

Figure 17. - Flow diagram of generator and synthesis-gas-purification train for powdered-fuel gasification in laboratory-scale pilot unit.