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<u>Coal name, number, and rank</u>	<u>Plastic properties</u>
Upper Elkhorn No. 3 slack (342) high-volatile A.	Showed evidence of being partly oxidized; maximum fluidity lower than same coal tested previously.
Blend of (342) with 35 percent Pocahontas (341).	Higher fluidity and lower maximum resistance than either of coals used.
Blend (342B) of 40 percent (342), 35 percent (341), and 25 percent Elkhorn egg (343) high-volatile A.	Slightly lower fluidity and appreciably higher maximum resistance than (342A).
Elkhorn egg (343) high-volatile A.	Typical of coals this rank; no resistance in Davis plastometer tests.
Corban (344) high-volatile A.	Similar to (343), except somewhat more fluid.
Blend (344A) of 65 percent Corban (344) and 35 percent Pocahontas (341).	Intermediate to those of its constituent coals; no resistance in Davis plastometer tests.
Blend (344B) of (344) 50 percent; (341) 35 percent; and (343) 15 percent.	Plastic properties similar to those of (344); no resistance in Davis plastometer tests.

#### Effect of Oxidation on Carbonizing Properties of Pocahontas Coal

The effects of oxidation upon the carbonizing properties of Pocahontas No. 3- and No. 4-bed<sup>87/</sup> coals were determined, and a second (pillar) sample from the No. 3 bed was studied to determine whether its coking properties had been altered by oxidation in place. No. 4 coal oxidized more rapidly and probably would lose its coking power first if both coals were stored under identical conditions. The pillar sample yielded 21 percent less tar than the tipple sample from the same bed, and tumbler tests showed that the coke from the pillar coal was more abradable. These results indicate that the pillar coal may have been oxidized enough to diminish its coking propensity.

#### Oxidizing Properties of Bituminous Coals

The effects of oxidation at room temperature and at 99.3° C. on 11 coking coals representing 9 beds were determined by agglutinating tests, chemical analyses, and carbonization tests at 800° C. of both fresh and oxidized samples.<sup>88/</sup> In general, continued oxidation of the coal decreased the agglutinating value, carbon and hydrogen contents, and heating value and increased the real specific gravity and oxygen

<sup>87/</sup> Davis, J. D., Reynolds, D. A., Brewer, R. E., Naugle, B. W., and Wolfson, D. E., Effect of Oxidation Upon the Carbonizing Properties of No. 3- and No. 4-Bed Coals from Eastgulf, Raleigh County, W. Va.: Bureau of Mines Tech. Paper 702, 1947, 18 pp.

<sup>88/</sup> Brewer, R. E., Reynolds, D. A., Steiner, W. A., and Van Gilder, R. D., Carbonizing Properties of Coking Coals, Effect of Oxidation in Storage: Ind. Eng. Chem., vol. 40, No. 7, 1948, pp. 1243-1254.

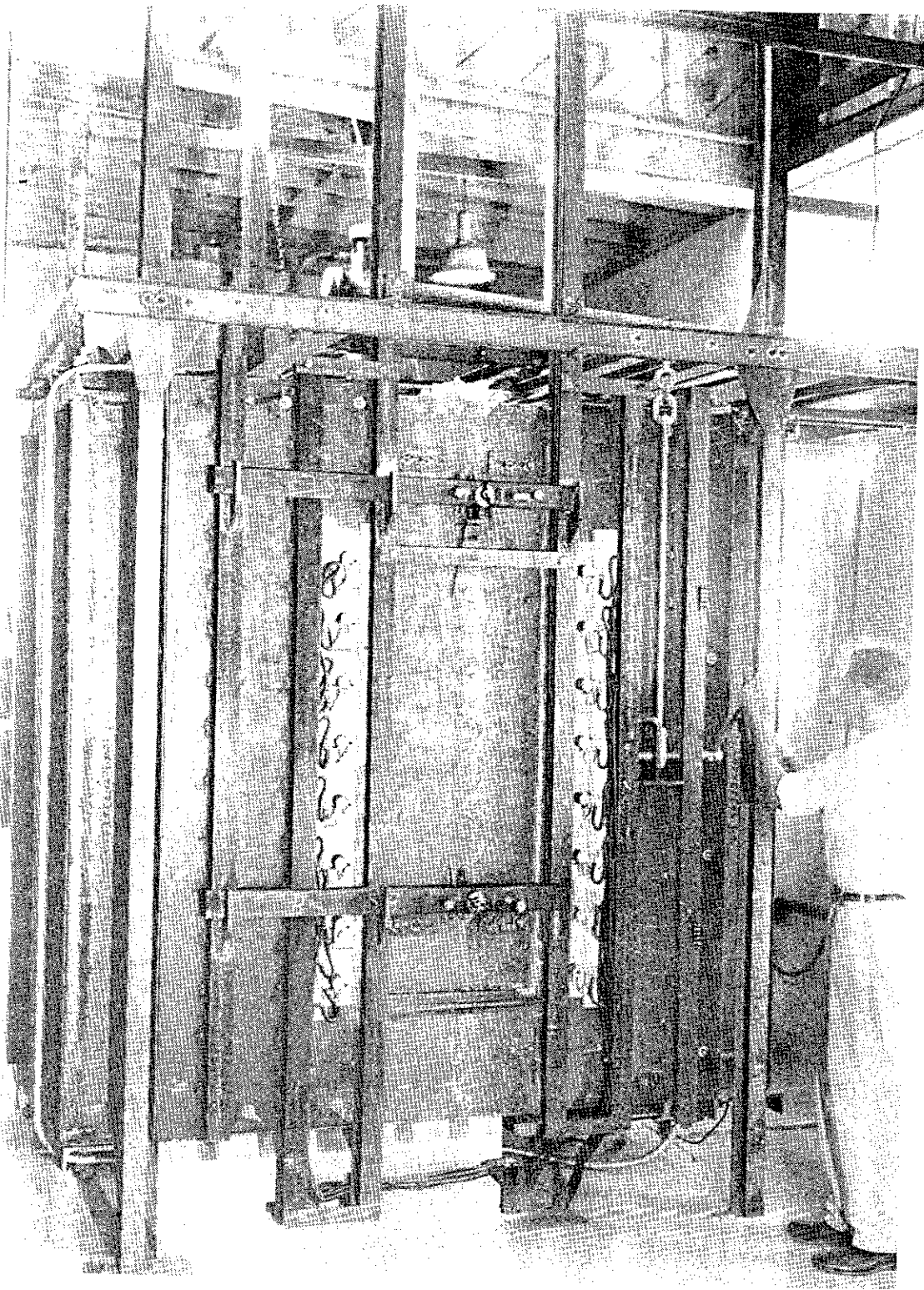


Figure 29. - Coke end of Bureau of Mines experimental coke oven.

content. The effects of oxidation on carbonizing properties were decreased strength and fusion of coke and yield of tar, increased apparent specific gravity of coke and yield of ammonium sulfate, and irregular proportions of acids and residual pitch in tar. The most sensitive measures of extent of oxidation of coal were agglutinating value, yield of tar, and coke-strength index.

Correlation of changes in coal and coke properties of the sample oxidized at room temperature with those of samples oxidized at 99.3° C. established simple factors that could be used to express relative rates of oxidation at the two temperatures. The number of days of oxidation at 99.3° C. required to decrease the coke-strength index by 15 percent, defined as durability of coking power, was selected as a basis for comparing the 11 coals. The durability of coking power at room temperature of each coal was computed from its durability at 99.3° C. by appropriate factors based on its agglutinating value and strength indexes of its cokes. The durability of the 11 coals, thus obtained, afforded a good indication of the known commercial storage behavior of the coals studied.

#### Desulfurization of Coal and Coke

Increased conversions of the sulfur in coal and coke samples to volatile sulfur products in the gas were obtained by carbonizing coals and reheating their cokes, first cooled to room temperature, in streams of added ammonia or hydrogen. Series of tests with and without passage of these gases at 700°, 800°, and 875° C., at room temperature, and at 500° C. were made on 15-gram samples of coal, sized to pass a No. 20 and be retained on a No. 40 U. S. sieve, and on their lump and crushed cokes. The desulfurizing action of ammonia gas, because of its almost complete decomposition into hydrogen and nitrogen at 800° C. or higher, was much more effective than that of hydrogen alone; for example, 67.2 and 48.8 percent, respectively, with lump coke at 800° C., as compared with 46.7 percent for Illinois No. 6 bed coal. This desulfurizing effect of ammonia was increased when added at lower temperatures - for example, 67.8 percent for ammonia at 500° C. and 59.2 percent for hydrogen at room temperature. Addition of ammonia gas at 800° C. to 0- to 20-mesh coke from a Pittsburgh-bed coal gave a conversion to volatile sulfur of 62.3 percent, as compared with 60.0 percent with the lump coke. Addition of nitrogen gas to coal and coke samples, because of its diluting action in the carbonization gases, showed lower conversions to volatile sulfur products than in similar tests without added gas.

Quantitative methods were developed for determining the volatile sulfur compounds - hydrogen sulfide, mercaptans, thiophenes, carbon disulfide, carbon oxysulfide, and residual sulfur - in the gas.

#### New Experimental Coke Oven

A new experimental coke oven designed and constructed at the Southern Experiment Station, Tuscaloosa, Ala., is shown in figure 29. Among the features of the new Bureau of Mines experimental oven, one or more of which are absent from all other known experimental ovens, are the following:

(1) Full-width coking chamber (17 inches). This eliminates the necessity of adjusting the experimental results for the estimated effects of thinner than normal charges.

(2) Heating walls are constructed of silica brick of average oven-wall thickness (4-1/2 inches). The new experimental oven, therefore, can be operated

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at any desired flue temperature and thus directly match the usual industrial practice in this respect. This feature eliminates the necessity for estimating temperature-progression curves for operation of the experimental oven and eliminates all errors arising from such estimates. Correlation between the quality of cokes produced in the experimental oven and those produced in full-scale ovens should be greatly simplified.

(3) Zoned heat-input arrangements are used so that the entire heating wall(s) can be maintained at any desired uniform temperature. The arrangements also permit temperatures to be varied from top to bottom, to make it possible to match the inherent characteristics of any particular battery of coke ovens, should such action be found desirable.

(4) The "movable" wall of the oven is suspended; the design is such that movements far in excess of those allowed during a coking test would require only a negligible force. (A pressure of 0.01 pound per square inch would move the unres-trained wall about one-eighth inch, whereas the maximum movement allowed under operating conditions is less than 0.02 inch.) All forces exerted by the coking charge, therefore, are actually transmitted to the recording mechanism and pressure losses due to friction or accidental blocking of moving parts are avoided.

(5) Pressure detecting and recording mechanisms are standard equipment purchased on the open market; no fine alinement or delicate adjustment is required to install or operate them.

(6) The oven is designed for coking pressures up to 10 pounds per square inch. This larger range of allowable coking pressure permits systematic investigation of the blending properties of coals and the underlying fundamentals over a wider range of blends than is possible with ovens having lower coking-pressure capacities.

Because of these and other features of design, the new oven will be useful in determining the significant effects of many known operating variables on the quality of coke. Among these, to name a few, which have not been satisfactorily investigated because of lack of suitable apparatus, are variations in flue temperature, moisture in charge, and tempering time.

However, these points of utility are in addition to the primary purpose of the oven - that is, to have available for use in determining the blending properties of coals, an experimental oven which is in all essential respects a small section of a modern industrial byproduct oven.

#### Low-Temperature Carbonization in Japan

The results of a field survey made of commercial low-temperature carbonization processes used in Japan before and during World War II, including a complete description of the processes employed and plants examined, have been published.<sup>89/</sup>

<sup>89/</sup> Reid, William T., Low-Temperature Carbonization of Coal in Japan: Bureau of Mines Inf. Circ. 7430, 1948, 82 pp.

## GASIFICATION

Gasification of Subbituminous Coal and Lignite

The results of investigations on gasification of low-rank coals from 1943-47 were analyzed and summarized.<sup>90</sup> Fundamental theories of gasification of coal in externally heated retorts were reviewed and the important aspects of heat transfer at high temperature were presented.

Analysis of data obtained on the large plant at Grand Forks, N. Dak., and the small pilot plant at Golden, Colo., indicated that further experimental work is necessary to improve operation of the annular retort. Some troubles were experienced in the large plant in the building up of ash deposit on the inner surface of the alloy retort when one grade of natural lignite was gasified, but these troubles were not noted in the small pilot plant when other grades of coal were gasified. The experimental data show that heat was transferred at a rate of about 5,000 B.t.u. per hour per square foot in the small pilot plant, corresponding to 70 to 80 cubic feet of water gas made per hour per square foot. The performance data on the pilot plants indicate that heat is transferred through the retort wall at over-all thermal efficiencies of about 60 percent. This is exceptional because the ordinary methods of heating retorts at high temperatures, employing conventional gas burners, indicate thermal efficiencies of only 30 percent. Because of the high heat-transfer efficiency attained in the retort developed by the Bureau of Mines, it is probable that such a heating system might be used effectively in the fluidized gasification of coal. Rates of heat transfer in the range of 10,000 B.t.u. per hour per square foot might be attained through the wall containing a fluid bed, and this would correspond to gas-production rates of 125 cubic feet per hour per square foot, which indicates that the 48-inch-diameter retort should have a production capacity of about 25,000 cubic feet of synthesis gas per hour. Such rates of production would make the process attractive for commercial production of water gas from subbituminous coal and lignite and should compete favorably with gasification systems using oxygen.

At Golden, Colo., limited experimental work was conducted on the gasification of coal and char dusts in suspension as a means of furnishing producer gas for heating the externally heated retorts. A small pilot plant of 4,000 cubic-feet-per-hour capacity was operated successfully on several tests.

Gasification of Lignite for Preparation of High-Hydrogen Water Gas

During the past fiscal year the Grand Forks, N. Dak., Pilot Plant produced a high-hydrogen water gas with a hydrogen : carbon monoxide ratio of 9. The plant was operated more than 1,600 hours, during which time some 570,000 pounds of lignite was processed and almost 13,000,000 cubic feet of gas made. This quantity of gas represents over 7,500,000 cubic feet of pure hydrogen, or between 25,000 and 35,000 cubic feet per ton of raw lignite gasified.

The gas by this process, particularly that with the higher hydrogen : carbon monoxide ratios, can be used directly for the partial or complete reduction of iron ores. The ore may be partly reduced to the magnetic oxide form for magnetic concentration, or it may be completely reduced to sponge iron or iron powder. After removal

<sup>90</sup> Parry, V. F., Wagner, E. O., Koth, A. W., and Goodman, J. E., Gasification of Subbituminous Coal and Lignite in Externally Heated Retorts; Ind. Eng. Chem., vol. 40, No. 4, 1948, pp. 627-641.



of carbon dioxide, the partly purified gases, particularly those with the lower hydrogen : carbon monoxide ratios, may be used as synthesis gases for the production of liquid fuels by the Fischer-Tropsch process or for the industrial preparation of synthetic organic compounds. By a further step, the carbon monoxide can be reacted with steam to obtain commercially pure hydrogen, which may be used in converting solid fuels into liquid fuels by the Bergius process, for production of ammonia and fertilizers, for hydrogenation of fats, and for other industrial uses.

#### Retort Corrosion During Gasification

The problem of retort corrosion during the gasification of lignite was analyzed. The economics of the process depends upon the life of the outer metal cylinder, through which flows the heat energy needed to maintain the reaction and which forms the outer wall of the reaction chamber. This cylinder is subject to severe operating conditions. It is maintained at a temperature between 1,400° and 2,100° F. At the same time, the outer surface of the cylinder is in contact with flame and combustion gases, while the inner surface is in contact with steam, hot carbon, and reducing gases, ash, sulfur compounds, and hydrocarbon vapors. The solution of this problem is imperative because a short retort-tube life will markedly increase the cost of gas production.

It was concluded that, as a result of more than 3,000 hours' operation, a Plura-melt outer cylinder (a metallic sandwich consisting of a 3/8-inch mild-steel center between two 1/8-inch layers of 26-percent chromium alloy) will give at least the expected life of 10,000 operating hours if the operating temperature does not exceed 1,900° F. if local superheating does not occur and if the oxidation caused by the reaction of excess steam with the chromium is reduced. It was determined further that, when using Pluramelt tubes, welds should be made with a 310 chrome-nickel alloy.

A similar investigation, based upon over 400 hours of operation, showed that "metcollized" metal was not a satisfactory material under these conditions.

These investigations are being extended to include other possible materials of construction.

#### Coal Investigations in Germany

Members of technical fuel missions to Germany organized under the auspices of the Technical Industrial Intelligence Committee have written a large number of reports describing German developments in mining, preparation, and utilization of coal. These reports are on file at the Bureau of Mines, Washington, D. C., where they are available for public examination. Because of the importance of the subject material, some of the reports or combination of reports were published.

Processing of high-temperature tar, utilization of the products obtained, and information concerning new and improved processes or products were obtained from visits to 40 targets in the American, British, French, and Russian zones of occupation.<sup>91/</sup> Certain gas-making processes used in Germany differ considerably from those in common use in America. Those investigated were<sup>92/</sup> the Lurgi pressure-gasification, Winkler, and Leuna slagging-type gas-producer processes, using steam

<sup>91/</sup> Rhodes, E. O., German High-Temperature Coal-Tar Industry: Bureau of Mines Inf. Circ. 7409, 1947, 117 pp.

<sup>92/</sup> Odell, William W., Gasification of Solid Fuels in Germany by the Lurgi, Winkler, and Leuna Slagging-Type Gas-Producer Processes: Bureau of Mines Inf. Circ. 7415, 1947, 46 pp.

and oxygen as the gas-making fluids. Observations were made on coking practice in Germany in preparation of metallurgical coke and the use of the sloping type of Didier gravity-discharging coke oven.<sup>93/</sup> The Pott-Broche process for extracting coal with solvents at elevated temperatures yields an extract containing only 0.05 percent ash. Distillation of the solvent and certain of the oils from the extract gives a residue that can be carbonized to produce a low-ash electrode carbon.<sup>94/</sup> Information on the organization of German fuel research and recent developments were obtained at six different research laboratories.<sup>95/</sup>

#### Underground Gasification

The underground gasification of coal is a process of exploitation of coal deposits through partial combustion by blasting with air, oxygen, steam, or other fluids and recovering fuel in gaseous form. Thus, the mechanical breaking down of the coal and its transportation to the surface are avoided. The advantages of such a process include the following: Possible utilization of coal beds too thin for economical mining; recovery of the fuel components in a form easily transported and efficiently utilized; avoidance of much difficult and dangerous labor; reduction of the man-hours required for production of a given quantity of fuel; and reduction of the cost of developing and operating a mine, especially where mining operations could be difficult.

The Bureau of Mines is attempting to determine the technical and economic factors involved in gasifying coal in place. Information is being obtained on: The thermodynamics and kinetics of the underground gasification reactions, necessary to determine the size of the installation required and the contact time required; the underground action of heat on overlying strata and underground heat losses; and the action of various gas making fluids and the quality and yield of products.

A laboratory-scale retort was built to simulate as nearly as possible some of the conditions encountered in actual underground gasification. The retort contained a bed of fine coal, about 14 by 7 feet horizontal dimensions; 1 foot 3 inches thick, and having an approximate capacity of 5,200 pounds of coal, enclosed in fire brick, well-insulated and the whole contained in a steel jacket open at the top. Twenty-four refractory tubes were installed at the bottom of the retort at various spots for gas sampling and measuring pressures and temperatures. Two rotary, lobe-type, blowers were installed, one for blast air, the other for removal of the product gas.

Seven experiments on this underground gasification retort have been conducted thus far, with varying conditions, such as length and type of channel, volume of air blown, and use of steam and regenerators. The regenerators were installed at the inlet and outlet of the channel after the second experiment.

Experiments were conducted with air flow through three loops of a "W"-shaped channel, and also other experiments with a single-loop "U"-shaped channel.

<sup>93/</sup> Reed, Frank H., Some Observations on Coking Practice in Germany. Part I. Metallurgical Coke, Part II. Slanting-Type Didier Coke Ovens, Stadtische Werke Karlsruhe: Bureau of Mines Inf. Circ. 7462, 1948, 74 pp.

<sup>94/</sup> Lowry, H. H., and Rose, H. J., Pott-Broche Coal-Extraction Process and Plant of Ruhrol G.m.b.H., Bottrop-Welheim, Germany: Bureau of Mines Inf. Circ. 7420, 1947, 12 pp.

<sup>95/</sup> Lowry, H. H., and Rose, H. J., Some Observations on German Coal Research and Developments: Bureau of Mines Inf. Circ. 7422, 1947, 27 pp.

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Typical data are given in table 17. Figures 30, 31, and 32 show the retort, roof, and residue after this same run.

TABLE 17. - Underground gasification retort run

Time from start to midpoint of cycle:	86.8	91.3	95.8	101.8	106.3	110.8	115.3	119.8	121.3	Average	Calculated
	hr.	hr.	hr.	hr.	hr.	hr.	hr.	hr.	hr.	age <sup>1/</sup>	air-free
Gas analysis, percent:											
CO <sub>2</sub> .....	11.5	10.5	9.2	9.0	8.8	8.6	8.3	8.9	8.6	8.8	8.9
Ill. ....	.6	.4	.5	.2	.4	.2	.2	.4	.0	.3	.3
O <sub>2</sub> .....	.0	.2	.0	.0	.7	.0	.3	.4	.0	.2	-
CO .....	13.6	15.1	18.0	18.5	19.9	19.1	21.1	19.7	21.3	19.7	19.9
H <sub>2</sub> .....	5.8	5.1	4.2	5.7	4.4	3.1	4.2	5.2	3.4	4.3	4.3
CH <sub>4</sub> .....	.9	.9	1.5	.6	.7	2.4	.7	.5	.9	1.0	1.0
N <sub>2</sub> .....	68.6	67.8	66.6	66.0	65.1	66.6	65.2	64.9	65.8	65.7	65.6
B.t.u./c.f. 60-30, dry .....	83.8	82.4	96.9	88.3	93.6	100.5	92.8	93.6	88.9	93.7	94.3
Average air flow <sup>1/</sup> .....	2,420 c.f.h.										
Average gas make flow <sup>1/</sup> .....	2,560 c.f.h.										
Cold-gas efficiency <sup>1/</sup> .....	55 percent										
Retort charge -	5,180 lb.										
Ultimate coal analysis as received:	Percent										
Hydrogen .....	4.6										
Carbon .....	75.9										
Nitrogen .....	1.2										
Oxygen .....	6.1										
Sulfur .....	.9										
Ash .....	11.3										
B.t.u. ....	13,220										

<sup>1/</sup> Average of last 7 analyses.

The laboratory experiments, while not conclusive, have demonstrated some interesting phenomena. Among these are:

A. The gas-making reaction begins in a short distance of blast travel (about 5 percent). The complete gas-making reaction apparently can be attained within the 75 percent of blast travel in the experimental retort.

B. The exploitation of a large area from an initial "U"-shaped passage may be very difficult. The tests indicated that a straight-line passage may be desirable.

C. Unidirectional blast flow is corollary to a straight-line passage. The advantages expected from unidirectional flow are: Simplified operation; less time required for warm-up; less capital cost in construction of hot-gas outlets; consistent product gas quality.



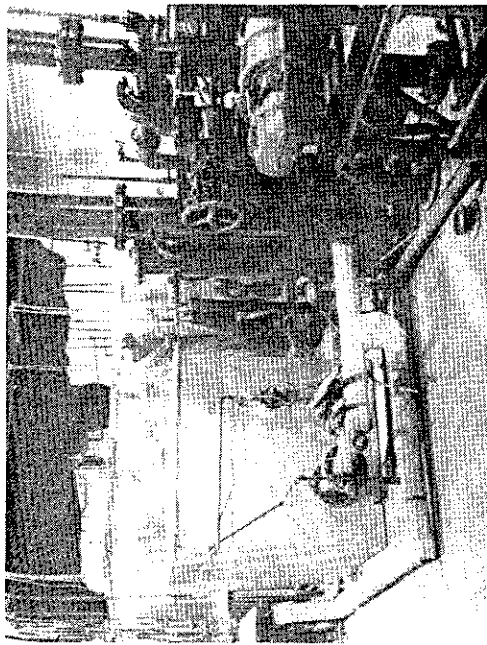


Figure 30. - Experimental underground gasification retort.

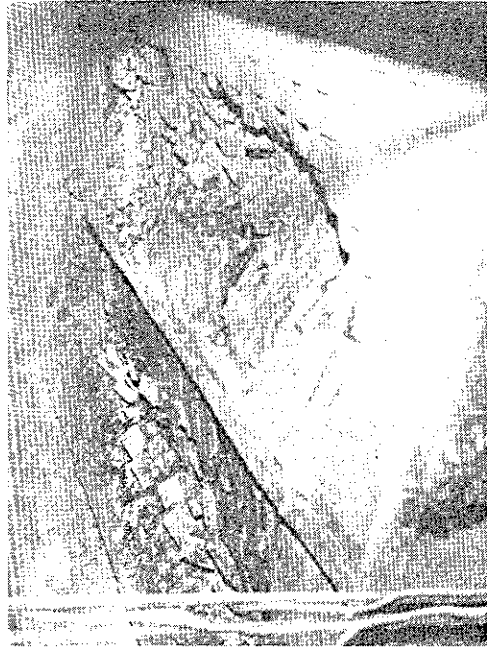


Figure 31. - Underground gasification retort; view of roof after run.



Figure 32. - Underground gasification retort; residue after run.

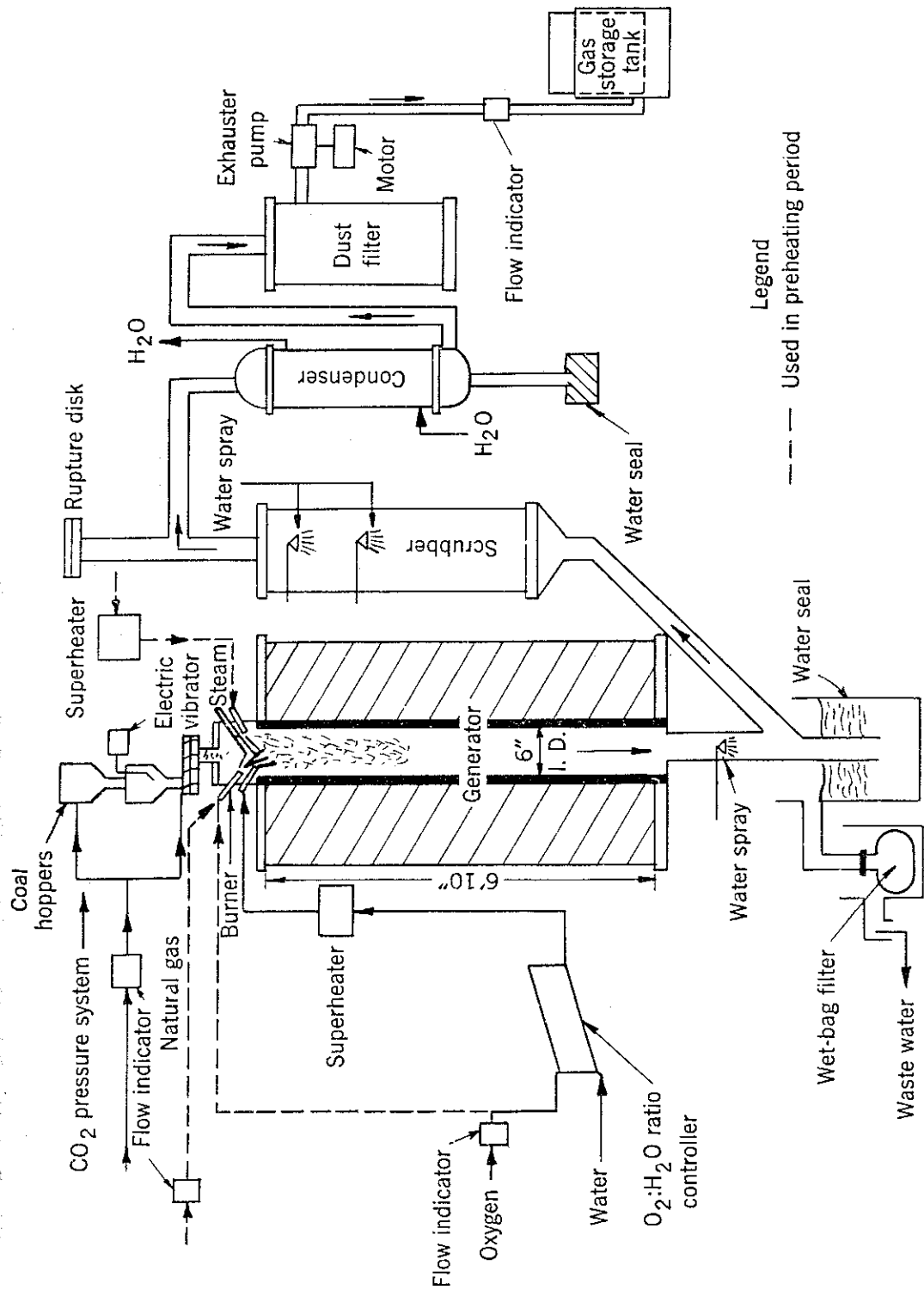


Figure 33. - Laboratory-scale apparatus for the gasification of pulverized fuel by entrainment in oxygen and steam. (Modified generator construction with straight tube arrangement; outlet at bottom; wet handling of products).

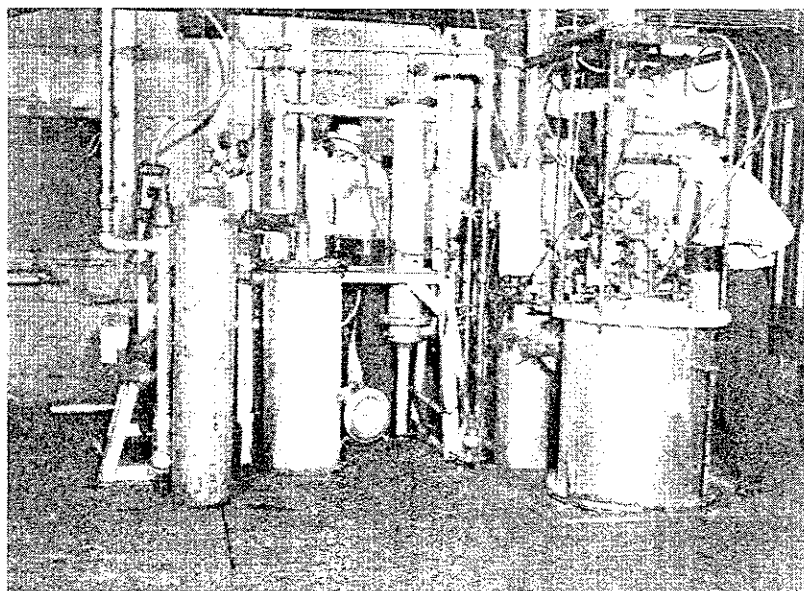


Figure 34. - Laboratory-scale gasification unit by entrainment of pulverized fuel in oxygen, showing accessory equipment.

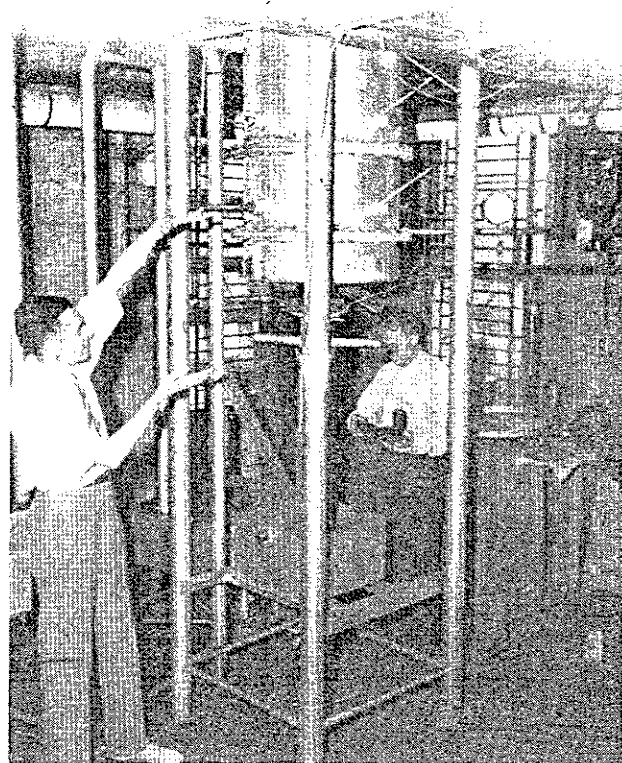


Figure 35. - Laboratory-scale gas generator; lower half.