

TABLE 4. - Typical performance of pressure gasifier operating at pressures of 100, 200, and 300 lb. per sq. in. on pulverized, Sewickley-bed coal

Run No.	34 A	34 B	34 C
Gasifier pressure p.s.i.g.	300	200	100
Duration of runhr.	3.00	3.00	3.00
Raw-coal ratelb./hr.	1,287	1,287	1,287
Process-steam inlet temperature.....°F.	905	900	900
Process-oxygen inlet temperaturedo.	30	30	26
Coal inlet temperaturedo.	250	240	240
Oxygen input per pound of coal std. cu. ft.	9.33	9.30	9.29
Steam input per pound of coal.....lb.	.30	.30	.30
Product-gas analysis (Correct for inert gas introduced with coal and at gasifier sight glass)..... percent:			
CO ₂	11.1	9.4	10.2
Illuminants.....	.5	.6	.4
H ₂	32.4	33.4	33.0
CO	52.5	53.5	53.1
CH ₄3	.2	.3
H ₂ +CO..... std.cu.ft./hr.	33,320	35,690	34,325
Total carbon gasifiedpercent	88.0	90.9	88.8
Coal required per M cu.ft. (H ₂ +CO).....lb.	38.6	36.1	37.5
Oxygen required per M cu.ft. (H ₂ +CO)..... std.cu.ft.	360	335	348
Process steam input per M cu. ft. (H ₂ +CO).....lb.	11.7	10.9	11.4
Coal throughput (hr.) (cu.ft.volume).....do.	674	674	674

After several preliminary runs, the reactor appeared as in figure 21. The slag deposition varied from 1/4-inch at the top where the flame temperature was most intense to 3/4-inch at the relatively cooler bottom. Depending upon such variables as oxygen-coal ratio, steam-coal ratio, and coal throughput, the heat losses per pound of coal varied between 800 to 1,600 B.t.u. The heat losses per square foot of reactor wall were in the range of 80,000 to 150,000 B.t.u., depending on operating conditions. The results show the feasibility of operation of a gasifier at 20 atmospheres with no refractories other than chilled slag. The effect of variations in reactor geometry and in throughput on heat loss per pound of coal remains to be determined.

The temperature of the cooling water passed into the coil in the reaction zone during the preliminary runs was 150° F. It may be possible to increase the temperature of the water and thereby reduce the loss of heat. Another possibility is to use, for the cooling coil, superior-grade steels of the type used on high-pressure and high-temperature boilers. In a coil of such material, steam may be safely generated, thus making possible a lower consumption of water and a lower heat loss to the coil.

Only minor modifications have been made to the remainder of the pilot plant (see figs. 22 and 23). Changes in line sizes made possible feeding of coal and other reactants at higher rates. The gas-metering system has been improved so that measurements are more accurate. Good results were obtained by scrubbing the gas with water under pressure (see table 4), using the scrubber as originally designed. Tests are being made using various depths of packing in the scrubber and various water rates per M cu. ft. of gas.

The slag-removal system has operated satisfactorily. Under most conditions, the slag and carbonaceous residue deposits in the base of the gasifier are in the form of very small particles which can be easily flushed out. Provision has been made to sample the gas as it leaves the reactant chamber. Initial tests indicate that the reaction is complete at this point.

Investigations of Coal-Feeding Methods

A pneumatic feeder is being used to feed the high-pressure gasifier. Efforts are being made to improve this unit to increase the intervals between charging the feeder with coal, to decrease coal carryover and thus improve recycle compressor operations, and to obtain better distribution of fluidizing gas.

Additional data are being obtained on the pressure drop in pneumatic coal-transfer lines. The largest size tube on which pressure-drop measurements have been made is 1/2-inch. To extend this range so that larger scale transfer lines can be designed, measurements will be made using a 1-1/4-inch schedule 40 pipe as the test section which will form a part of the regular coal-transfer system. The flow of coal will be from the fluidized feeder back to the transfer hopper, which is mounted on scales. The pressure drop in a straight-line section and in a standard 90° bend will be measured at pressure levels between 50 and 250 p.s.i.g.

A system for feeding coal into a pressurized reactor without the gas loss inherent in any type of lock hopper is especially attractive. Tests have shown that slurries of approximately 50 percent water and 50 percent coal by weight can be pumped up to pressures of 500 p.s.i.g. This was accomplished by the use of a reciprocating pump with ball valves. A system has been designed for mixing, pumping, vaporizing, and superheating 1,600 pounds per hour of a slurry composed of equal parts of coal and water, and for feeding the final mixture of superheated steam and coal into a pressure gasifier operating at 450 p.s.i.g. The temperature of the mixture going into the gasifier will be 800° F. or lower. The system requires the following equipment:

1. Slurry Mix Tank. - Complete with turbomixer for obtaining intimate contact between the coal and water. The capacity of this mixing tank at any one time is 1 ton of slurry.
2. Slurry Pump. - This is a standard commercial duplex reciprocating pump. The output of the pump may be varied between 1 to 5 gallons of slurry per minute. The output pressure of the pump is 600 p.s.i.g. The pistons and ball-type valves are made of 18-8 stainless steel. Because the slurry contains 1 pound of water per pound of coal and only 0.3 pound of water per pound of coal is required as steam for gasification, some feasible way must be found for separating the excess superheated steam from the coal. Available commercial mechanical separators do not produce steam with a low enough content of solids.
3. Slurry Heater. - This device resembles a monotube boiler and is a continuous coil wound in the form of two concentric helices. The coil, heated by a natural gas-fired burner, is made up of 530 lineal feet of 1-inch schedule 80 pipe installed in a refractory-lined shell. The heater is designed for an overall coefficient of 10,000 B.t.u. per hour per square foot of inside coil diameter and should heat 1,600 pounds per hour of 1 : 1 slurry at a pressure of 450 p.s.i.g. to a temperature of 800° F. The flowsheet for this system is shown in figure 24.

Using unheated equipment, various type valves are being tested for use as pressure control valves and as shut off valves. Orifices are being used to aid in

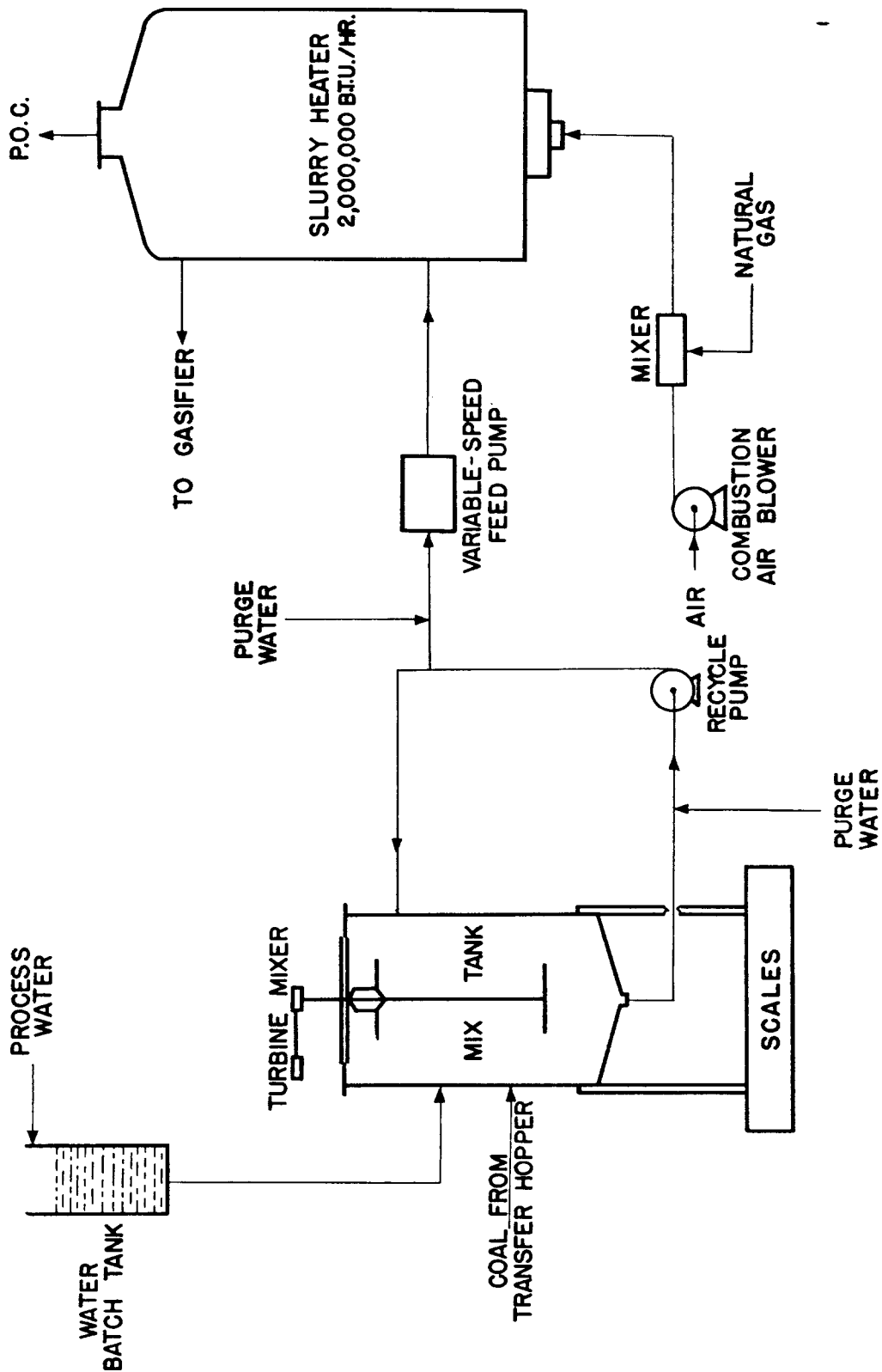


Figure 24. - Simplified flowsheet for slurry system.

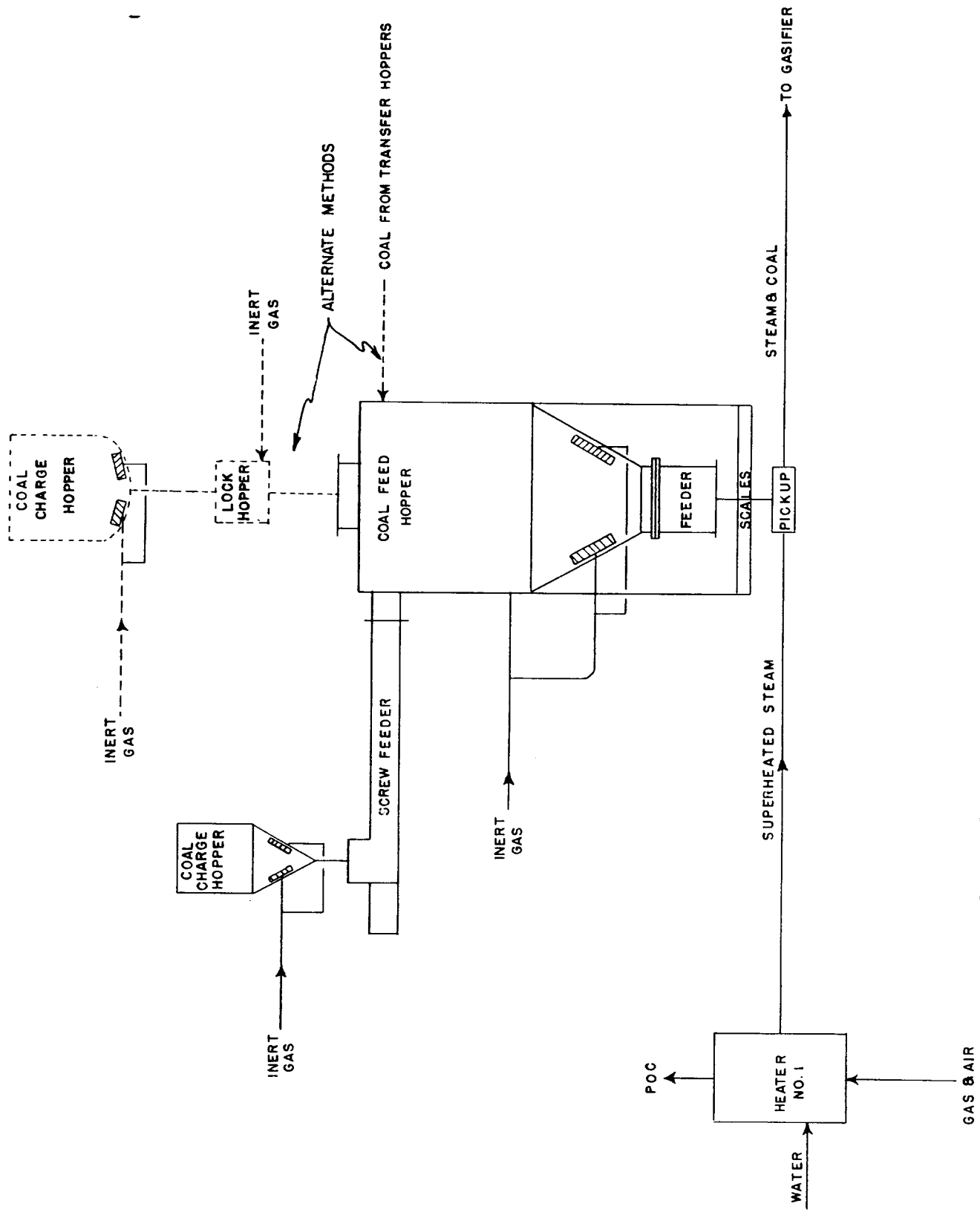


Figure 25. - Simplified flowsheet for steam pickup system.

establishing a high pressure in the pump discharge line to simulate conditions to be encountered when pumping the mixture into a pressure vessel. Long pumping tests are being made to determine rates of wear of equipment.

Feeding Coal Into Pressurized Vessels

A special screw feeder with interrupted vanes of a type that has been used to feed cottonseed meal into pressurized vessels has been acquired at Morgantown and will be tested for feeding coal into storage tanks at pressures up to 30 atmospheres. Another system being studied consists of a pressurized hopper from which the coal will be fed into a stream of superheated steam that will convey the coal into the gasifier. This system is illustrated by the flowsheet in figure 25. A star-wheel feeder with a casing suitable for 500 p.s.i.g. has been ordered.

Injection-Burner Investigation

The action of the coal-gas mixture as it leaves the injection burners is being studied. The ultimate aim is to obtain information on the spread of the mixture as it leaves the burner, both in cold test and as fired. The information will be used in designing burners to provide good mixing with as low an inlet pressure of oxygen as possible. Some savings in operating costs could be effected by lowering the presently high pressure of oxygen at the inlet. A chamber with plastic windows is used for the cold tests. Air is used in these tests to simulate the flow of steam plus oxygen through the burner nozzles. The burners will be lighted in a special atmospheric chamber or in free space to obtain data on the behavior of the flame. The coal is fed to the burner from the low-pressure fluidized-coal feeders used for gasification runs.

Oxygen Plant

The oxygen plant described in the 1952 Annual Report was operated during the past year. A substantial saving was effected in oxygen cost for operating the gasification pilot plants. Arrangements are being completed to purchase the plant from the Air Products Co., Inc. Besides supplying over 7,000 M cu. ft. per hr. of oxygen for the pilot plants and all of the oxygen needed for plant maintenance, the plant supplies nitrogen which is used to supplement the supply of inert gas for use in purging equipment. Perhaps the greatest value the plant has lies in the fact that it makes possible longer sustained tests in the gasification pilot plants.

High-Ratio Centrifugal Compressor

In collaboration with a commercial manufacturer of compressors a high-ratio, low-flow centrifugal compressor has been designed, and fabrication is 70 percent complete. Its capacity is 1,000 std. cu. ft. of air per minute at 120 p.s.i.g. The compressor has 2 single-stage impellers and is driven by a 200-hp. induction motor. Unloading and speed control are by means of an induction clutch. The use of two single-stage wheels offers a considerable gain in efficiency over the conventional-type machine, in which several impellers are mounted on the same shaft. In addition to furnishing pulsationless, oil-free air it is hoped that the machine will be a prototype for large-scale oxygen compression. A high-efficiency centrifugal compressor is needed for air compression in oxygen plants operating on the Linde-Frankl cycle. It also has a place as a synthesis-gas booster and for recycling. The low weight-power ratio of the centrifugal compressor should be advantageous for furnishing compressed air for the mining industry. The small foundation required should permit moving the compressor as mining operations progress. This movability saves air piping and reduces loss of pressure.

Laboratory-Scale Experimentation

Steam-Carbon Reaction Rate

The rate of the steam-carbon reaction at relatively high temperatures is being studied. The only data now available on the rate of this reaction at 2,500° F. were obtained using either a bed of carbon particles or a carbon rod. These studies were made under conditions far different from those in a pulverized-coal gasifier. Data will be obtained on the rate of the steam-carbon reaction in the temperature range of 2,000° to 2,500° F., using a system more closely approaching the conditions in the gasifier in which small carbon particles are suspended in the gas and in which there is a deficiency of carbon. Small particles of carbon will be dropped through steam at several temperatures. Sufficient excess of steam will keep the gas composition essentially constant. Residence time will be varied to determine the initial rate. The extent of reaction will be determined by measuring the weight of carbon consumed.

The apparatus consists of an electrically heated, vertical, cylindrical furnace 3 inches I.D. and 6-1/2 ft. long. The carbon particles are dropped through a 5-mm. tube at the axis of the cylinder and extending about 2 feet down from the top. Pre-heated steam enters the top of the reactor through a distribution ring and flows down through straightening vanes and contacts the carbon at the end of the carbon-inlet tube. Carbon and steam flow in parallel to the furnace exit, where a water spray quenches the reaction and condenses the unused steam. Unconverted carbon is collected in a vacuum filter directly below the reactor.

Gas Purification

Analytical Methods

A method was developed for determining carbonyl sulfide in synthesis gas. It is based on hydrolysis of this compound in dilute potassium hydroxide and determination of the sulfide ion, using the methylene-blue test. A sample of test gas is drawn into a glass container by means of a measured amount of the absorbent (0.1N KOH) in a leveling bottle, and the container is shaken for 20 minutes. The absorbent is then removed and subjected to the methylene-blue test. Hydrogen sulfide interferes with the test and must be removed. Carbon dioxide in concentrations above 4 percent also interferes, but this difficulty can be eliminated by first bubbling the gas through 40 percent KOH. The method is especially useful in determining the concentration of carbonyl sulfide in synthesis gas containing other common organic sulfur compounds. Carbon disulfide, ethyl mercaptan, and thiophene do not interfere with the test.

Studies of procedures for measuring the amount of dust in raw and purified synthesis gas were continued. Particle examination and size determination by microscopic methods were hindered by lack of satisfactory methods for obtaining a dry dispersion of particles. A satisfactory method was finally developed by dispersing the particles in benzene and drying by means of a controlled air flow (see fig. 26). Microscopic examination of particles in synthesis gas leaving the gasifiers has shown some interesting facts (see fig. 27). The ring formations are quite common and may be caused by vortices. The vents caused by escape of volatile matter are clearly shown.

As the size of the dust agglomerates actually existing in the gas stream is more important than the ultimate particle size, methods for their determination have been studied. Certain optical methods are theoretically possible, and sedimentation method has been developed that appears satisfactory, at least for particles larger than 10 microns in diameter.

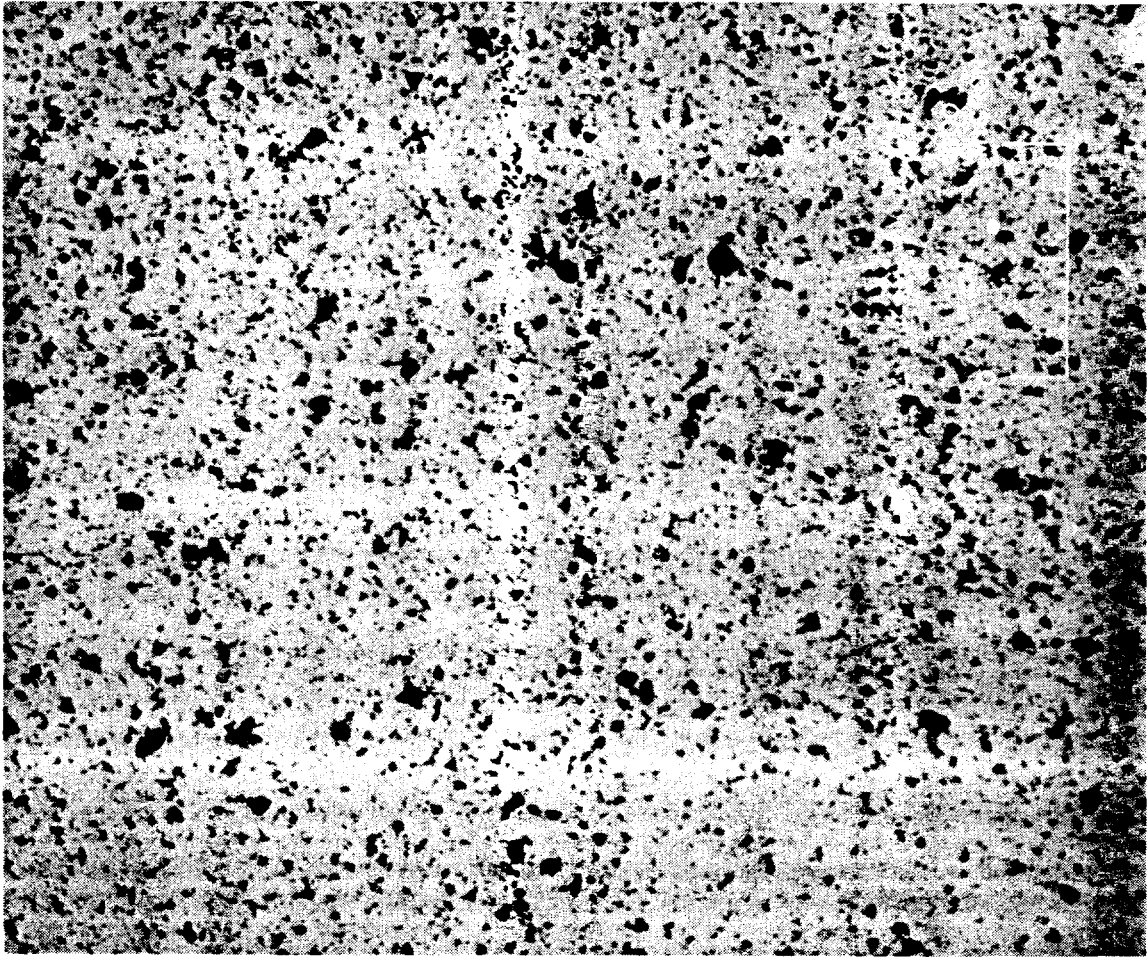


Figure 26. - Typical dust dispersion in synthesis gas.

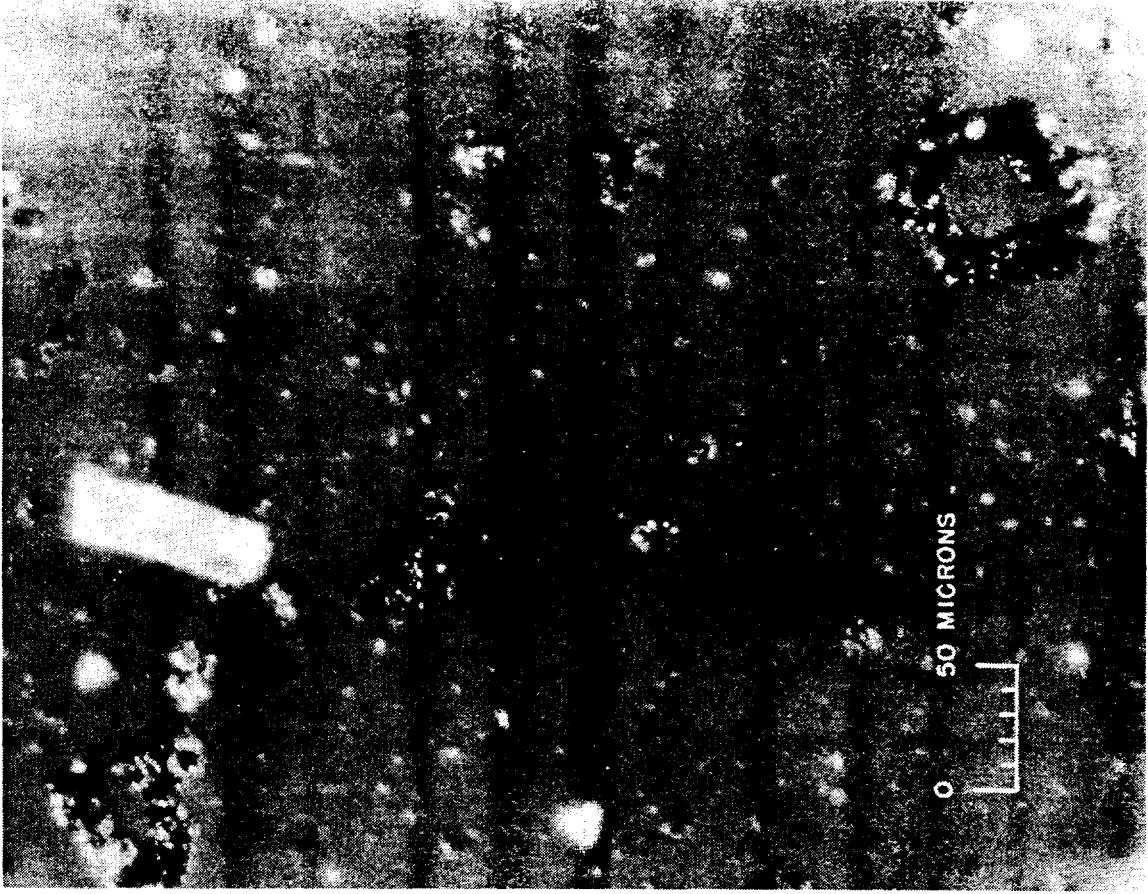


Figure 27. - Magnified residue particles in gas leaving atmospheric-pressure gasifier.

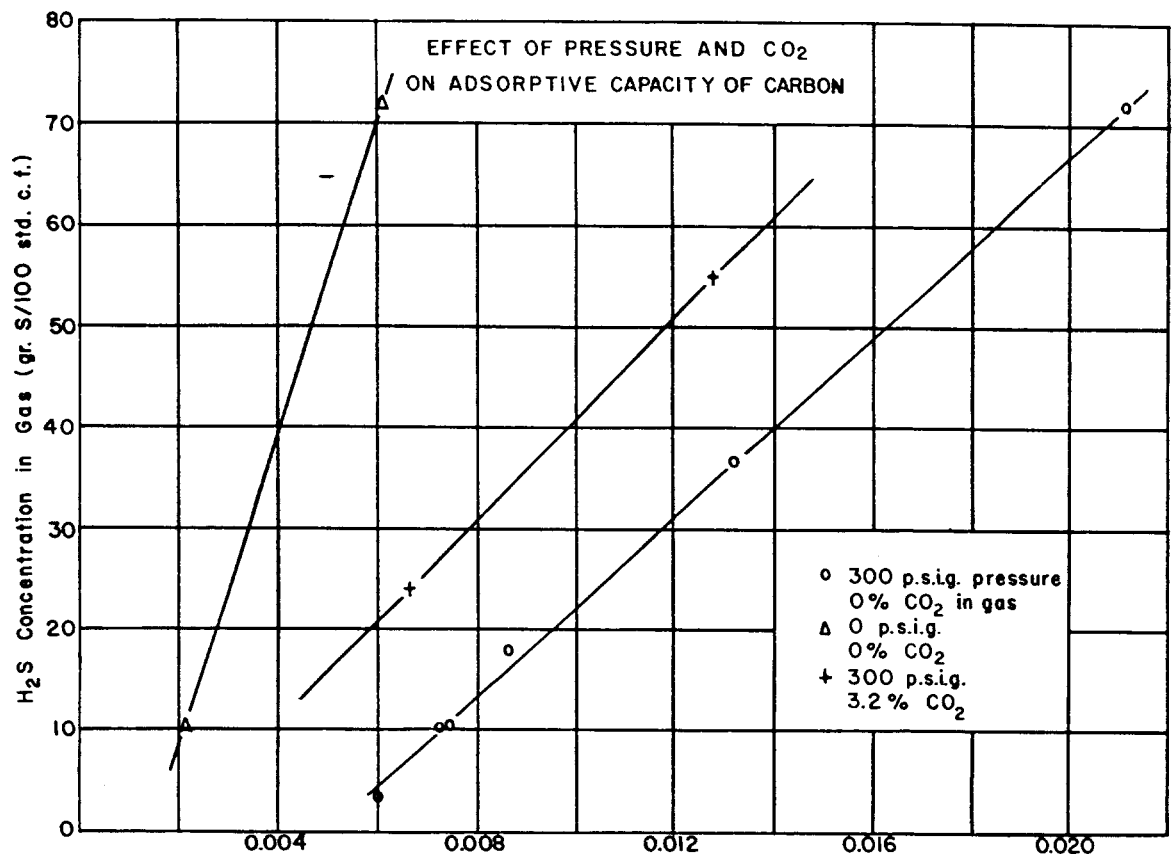


Figure 28. - Effect of pressure and carbon dioxide on adsorptive capacity of activated carbon for varying hydrogen sulfide concentrations.

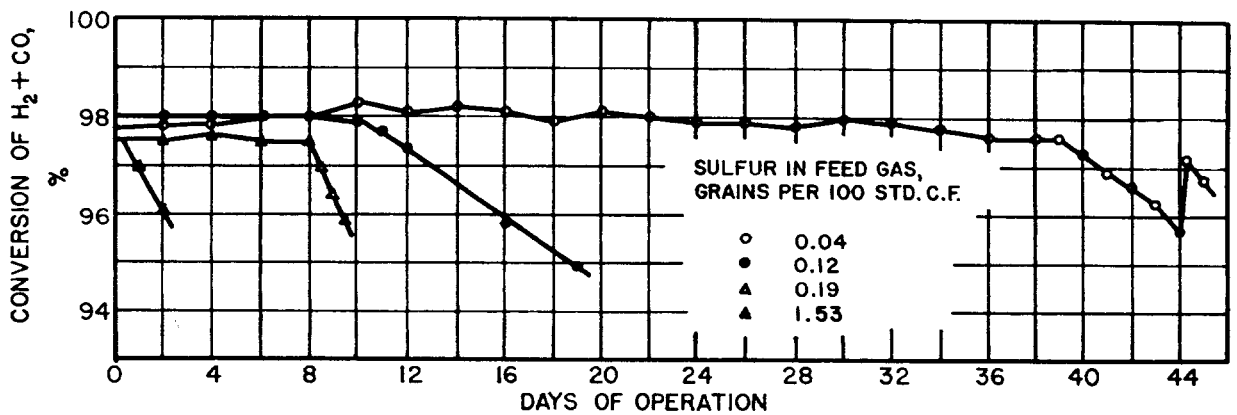
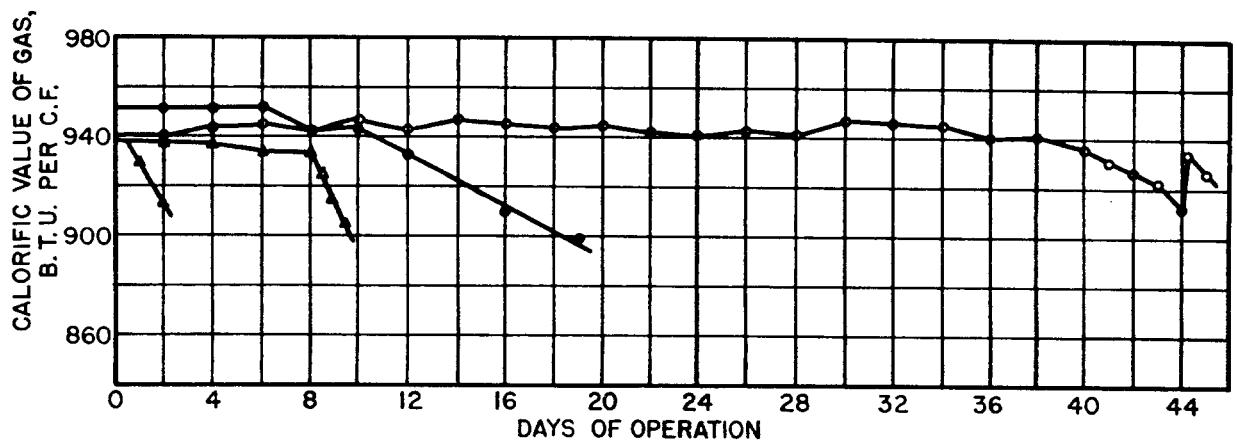


Figure 29. - Variations in hydrogen and carbon monoxide conversion and calorific value of product gas for methane-synthesis tests.

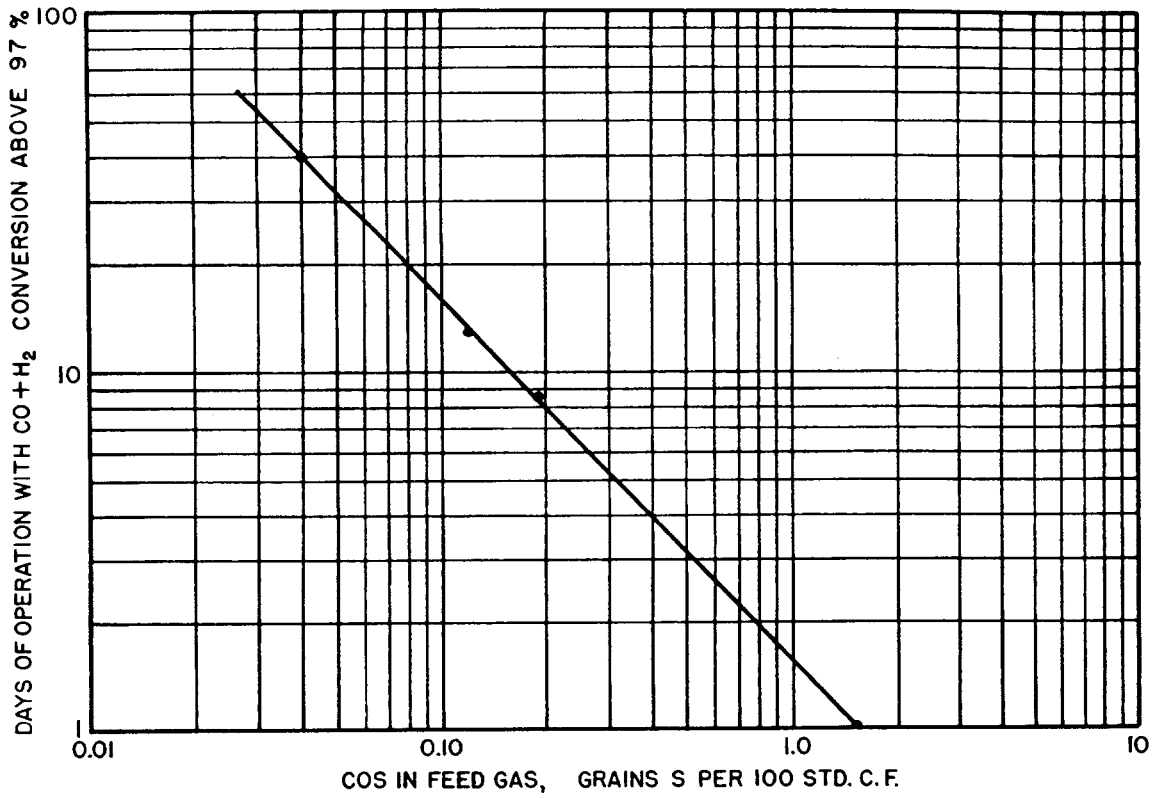


Figure 30. - Effect of sulfur content of feed gas on catalyst activity.

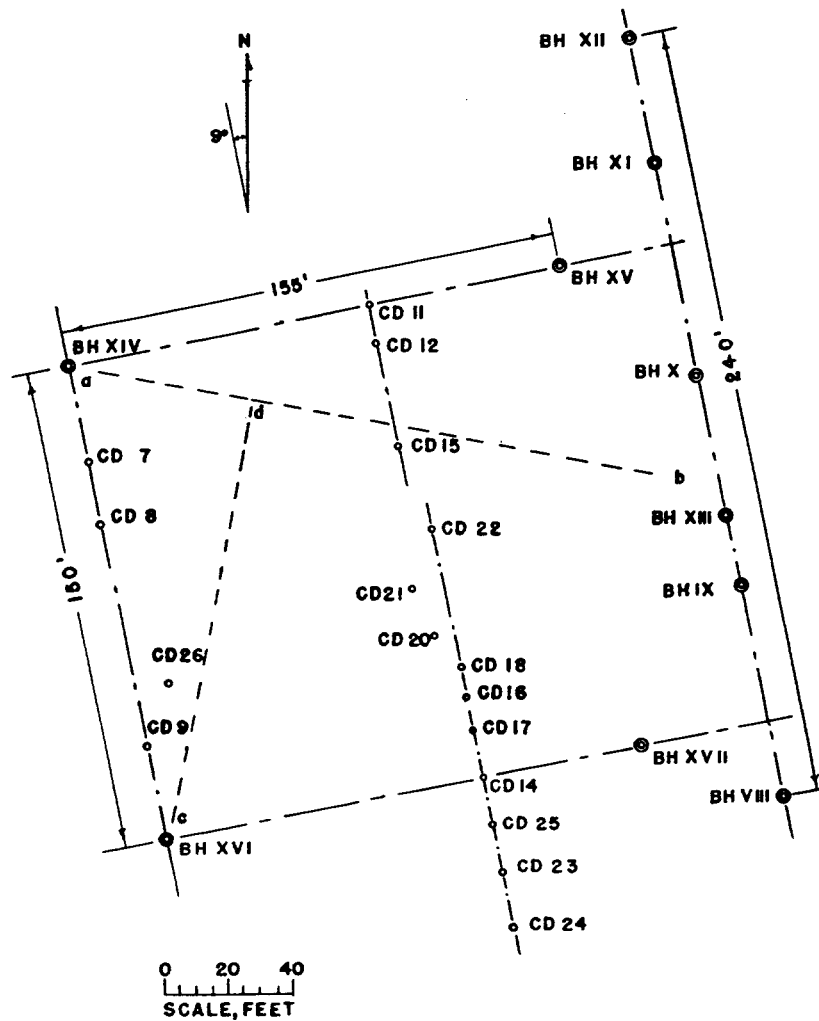


Figure 31. - Plan of electrolinking gasification trials.

Bench-Scale Experiments

It has been shown that a purified synthesis gas meeting specifications for the Fischer-Tropsch synthesis can be obtained in a three-step process: (1) Removal of the carbon dioxide and the bulk of the hydrogen sulfide by scrubbing with an aqueous solution of diethanolamine; (2) removal of the residual hydrogen sulfide by iron oxide purification; and (3) adsorption of organic sulfur compounds by activated carbon. A saving in purification costs could be realized if the iron oxide purifiers could be eliminated and the residual hydrogen sulfide removed simultaneously with the organic sulfur by activated carbon. Accordingly experiments were made at atmospheric and 300 p.s.i.g., using activated carbon to remove hydrogen sulfide in varying concentrations from a stream of hydrogen containing 3.2 percent and no carbon dioxide.

The tube containing the carbon was made of 1/2-inch, type-304, stainless-steel, schedule 40 pipe. The overall height of the adsorber was 48 inches and most of the bed depths were 9 to 10 inches. Hydrogen sulfide determinations were made on the gas entering and leaving the bed of carbon. The adsorptive capacity of the carbon varies linearly with the concentration of the hydrogen sulfide in the influent gas, for the range between 10 and 72 grains per 100 std. cu. ft. of gas (see fig. 28). The effect of pressure and carbon dioxide also can be noted. The curves are, in effect, equilibrium curves for the conditions shown.

Experiments were continued under a cooperative agreement with the Southern Natural Gas Co. and Tennessee Gas Transmission Co. for studying the catalytic conversion of synthesis gas to methane, and more specifically to determine the effect of sulfur on the activity of a nickel catalyst. Five runs were made using simulated Lurgi gas^{3/} having a hydrogen-carbon monoxide ratio of about 2:1 and sulfur concentration (as COS) ranging from nil to 1.53 grains per 100 std. cu. ft. Figure 29 summarizes the results from four runs by showing from day to day, the percent conversion of hydrogen and carbon monoxide and the calorific value of the product gas. The calorific value shown is the gross heating value based on dry gas measured at 60° F. and 30 inches of mercury, assuming a carbon dioxide-free gas containing 3 percent nitrogen. The results show that the sulfur concentration in the gas, multiplied by the number of days of operation during which the conversion of hydrogen and carbon monoxide was above 97 percent, is a constant (see fig. 30). With a gas containing 0.04 grain of sulfur as carbonyl sulfide per 100 std. cu. ft., the conversion of hydrogen and carbon monoxide remained above 97 percent for 40 days. During that time, the heating value of the product gas (on a CO₂-free basis) remained relatively constant at 940 B.t.u. per cu. ft.

In all runs in which sulfur was present in the gas, catalyst activity decreased as soon as approximately 0.58 gram per 100 grams of nickel had been absorbed. An attempt was made to regenerate the spent catalyst from 1 run by burning off the sulfur with air at 900° C. When reused, the regenerated catalyst gave an initial conversion of hydrogen and carbon monoxide of 91.8 percent.

A study was made of the moving-bed filter for removing dust from gas. The gas flows upward through a tower filled with granular material that is continually moved downward. The filtering material is discharged from the bottom, washed, and returned to the top of the bed. Any desired degree of dust removal can be obtained by using finer filtering material or by increasing the amount of dust in the bed by decreasing the rate of recycle of the granules. This method was used on a large scale in Germany and was considered more satisfactory than the electrical precipitator or Theisen disintegrator.

^{3/} Gas similar to that produced from coal by a Lurgi gasifier operated at about 20 atmospheres pressure.

Experiments on the moving-bed filter showed:

1. The percentage of dust removal increases rapidly as the pressure drop is permitted to increase by allowing the amount of dust in the bed to build up. This continues until the dust layer reaches the top of the bed, after which increased pressure drop gives lower dust removal.
2. Deeper beds give better removal at the same pressure drop.
3. At constant flow of gas, better removal is obtained when wet rather than dry coke is used, but the pressure drop is much higher when wet coke is used. However, at the same pressure drop, better removal is obtained with dry coke, probably because the water seals the pores in the coke. This conclusion is based on experiments which showed that, with smooth filtering materials, such as alumina or mullite spheres, much lower removal efficiencies are obtained than with rough materials such as coke. To obtain the same pressure drop with wet beds, a higher recycle rate is necessary than with dry beds.
4. Removal efficiencies of over 99 percent were obtained with pressure drops of 2 to 6 inches of water.

Pilot-Plant Operation

The moving-bed coke filter, when used on gasification system 4 following the water scrubber, reduced the dust concentration from 30 to 1.5 grain per 100 std. cu. ft., a removal of 95 percent. This efficiency was obtained with a pressure drop of a few inches of water. Tests at pressure drops of 12 inches of water reduced the dust concentration from 40 grains to 0.2 grain per 100 std. cu. ft. This equals the results ordinarily obtained with the electrical precipitator in the system.

A study has been made of two sets of scrubbing equipment used in the pilot-plant purification systems to determine their effectiveness. The unpacked fogging chamber following the atmospheric pressure gasifier 4 was designed to reduce the temperature of the gas sufficiently for the use of unlined steel pipe, but it also removes about half of the dust in the gas leaving the gasifier. The packed scrubber following the fogging chamber reduces the dust concentration from several thousand grains per 100 std. cu. ft. to about 30 grains per 100 std. cu. ft. Pressure is the most important variable in water-scrubber performance, as shown in table 5. This table presents results obtained with the high pressure scrubber on gasification system No. 3.

TABLE 5. - Dust concentration in gas leaving pressure scrubber

<u>Pressure p.s.i.g.</u>	<u>Dust concentration, grains per 100 std. cu. ft.</u>
100	4.5
200	.90
300	.65
450	.25

The decrease in dust concentration is approximately proportional to the square of the pressure. At 100 p.s.i.g., the dust concentration is 34 grains per 100 cu. ft. at the existing pressure, while at 450 p.s.i.g. the dust concentration is 7.8 grains. The effect of water rate is also noticeable, dropping the dust concentration from 1.7 grains per 100 std. cu. ft. with 80 pounds of water per 1,000 std. cu.

ft. to 0.6 grain per 100 std. cu. ft. with 200 pounds of water per 1,000 std. cu. ft. Over 50 percent of the weight of the dust leaving the gasifier and fogging chamber is greater than 44 microns in diameter, while more than 50 percent of that leaving the scrubber is over 20 microns and that leaving the moving bed filter is over 10 microns.

UNDERGROUND GASIFICATION PROJECT, GORGAS, ALA.

Since 1946 the Bureau of Mines and the Alabama Power Co. have conducted field-scale experiments on underground gasification of coal. The investigation was to determine the feasibility of procedures whereby either the chemical constituents or the energy of the coal may be brought to the surface in a form usable in the synthesis of liquid fuels or in the production of electric power.

In the first experiments, passages were driven into the coal bed by mining, and gasification was attempted by igniting the coal and passing the gasmaking fluids through these passages. Since 1951 electrolinking-carbonization has been substituted for underground mining in preparation of the site. Electrodes were inserted in two vertical holes drilled to the coal bed, and an electric current was passed between the electrodes in contact with the coal. The passage of the electric current caused carbonization of a quantity of coal, and the fissures formed during carbonization permitted the flow of gasmaking fluids during the gasification operations that followed. The electric current was used only during the site preparation phase and was discontinued during gasification. The electrolinking-carbonization technique was originally developed by the Sinclair Coal Co., working in cooperation with the Missouri School of Mines. It was used at Gorgas under a cooperative agreement between the Bureau of Mines and the Sinclair Coal Co.

In December 1952, experiments were completed according to the plan shown in figure 31, after which the underground reaction zones were then flooded. By the end of February 1953, the fires had been extinguished, and much of the heat stored underground had been dissipated. Thus far, there are no indications that combustion has started again.

The Bureau of Mines entered into a cooperative contract with the Stanolind Oil & Gas Co., and the Halliburton Oil Well Cementing Co., to try a hydraulic method of fracturing underground strata to prepare a passage in the coal bed. This system has been applied in oilfield work and is known commercially as Hydrafrac. Installation of equipment and preparation of the site for this test were approximately two-thirds complete by May 1. At that time it was necessary to terminate all work at the project and place the equipment in standby condition, as appropriations for the fiscal year beginning July 1 did not include funds for underground gasification.

In applying electrolinking-carbonization to prepare the site, it was expected that the current would flow by the shortest linear path between the electrodes. The first linking trials were made along the line boreholes VIII to XI (see fig. 31). These trials were not fully successful because of improper electrode installation; however, a linkage was accomplished between boreholes VIII and IX, a distance of 67 feet. This path was then gasified with air. A second and successful linkage was then made between boreholes XII and XIII, a distance of 152 feet. This path also was gasified with air. In these trials, the flow of current was substantially in the direction of the electrodes, but the following gasification operations were along the line of the boreholes. After gasification, the "burned-out" region was surrounded by a layer of coke. A third successful linking was then made between boreholes XIV and XV. It was expected that the path again would be substantially a straight line between electrodes, although it was realized that presence of the coke

boundary surrounding the previous workings could act as a very large electrode and cause distortion of the path. A fourth successful linking was made between boreholes XVI and XVII. Again it was expected the path would be substantially a straight line between electrodes; but, as before, the coke paths existing underground might distort the direction. Power usage during the 3 linking trials differed greatly; the path XII-XIII required 40,500 Kw.-hr., to effect linkage, the path XIV - XV required 146,800 Kw.-hr., and the path XVI - XVII required 20,080 Kw.-hr. These power requirements indicated widely different conditions underground and possibly the coke boundaries were responsible.

When fieldwork was discontinued in May, the direction of the paths underground had not been determined accurately. Test holes had been drilled at CD 11, 12 and 14 (see fig. 31), and none of these intersected the reaction zone underground. Test hole CD 15 intersected a "burned-out" region. Since June 20 the Alabama Power Co. has maintained a small crew at the project, and in August a drilling survey was begun under the direction of the Bureau of Mines. Test holes were drilled at locations CD 16 to CD 25, inclusive, without making contact with the underground reaction zones. Test hole CD 26 intersected the "burned-out" region. Additional test holes were planned to determine the direction, extent, and, if possible, something of the nature of the material in the reaction zones underground. Incomplete data indicate that the paths follow the dotted lines ab and cd, (see fig. 31). The coke boundary around the reaction path between boreholes VIII and XII probably was at least partly responsible for deflecting the path originating at borehole XIV (as line ab). This may account in part for the high power requirements for effecting linkage. The path (cd) originating at borehole XVI apparently was deflected approximately 70° and probably intersects the path (ab) originating at borehole XIV. The shorter distance may account for the relatively low power requirement to effect linkage along this path. Spacing of test holes CD 16 to 25 is such that, considering the tonnage of coal utilized, it is virtually impossible for the path to have passed between adjacent holes.

Several methods of gasmaking were tried during the gasification phase of operations in which electrolinking-carbonization was used to establish the initial path. Using an air blast entirely, boreholes XIV to XV were operated as one unit, discharging into the old system boreholes VIII to XII. Another unit, boreholes XVI to XVII, also discharging into the same boreholes, was operated in various ways, including continuous air blast, continuous oxygen blast, air blow followed by steam run, air blow followed by a mixed air-steam run, and air blow followed by a run using a reduced air blow. These methods were tried to obtain qualitative information on the types of gas that could be produced by underground gasification. Table 6 summarizes the operating data. In each instance the results are the average either for an extended period of continuous operation, or for a number of similar and consecutive cycles. The composition shown for the gas made by cyclic operation is the weighted average obtained by integration of the composition-time curves. As this method requires constant product gas flow throughout each cycle, steady operation was substantially maintained. During many steam runs the heating value of the product gas reached 300 B.t.u. per cubic foot, and the hydrogen content exceeded 50 percent.

Most of the experiments conducted at Gorgas were done with a continuous air blast; the various other types of operation represent first tests. In the cyclic operation, the blast and the run were always made in the same direction, and cycle times were selected arbitrarily. These conditions do not necessarily represent the optimum.

TABLE 6. - Experimental operating results obtained using various types of gasmaking fluids

Column	1	2	3	4	5	6	7	8
Type of operation	Continuous	Continuous	Cyclic	Cyclic	Cyclic	Cyclic	Cyclic	Cyclic
Gasmaking fluid:								
a. Blast	Air	Oxygen	Air	Air	Air	Air	Air	Air
b. Run	-	-	Steam	Steam	Steam	Air/steam	Air/steam	Air (reduced flow)
Cycle time.....hours:								
a. Blast			18.8	6	6	19.3	32	18
b. Run			4.2	6	6	4.8	4.9	6
Relative age of unit.....percent ^{1/}	9-71	34-43	47-67	89-94	97-99.5	78-83	83-89	71-78
Unit life represented.....do. ^{1/}	62	9	20	5	2.5	5	6	7
Input:								
a. Blast, air....cu.ft.per min. ^{2/}			1,250	2,520	2,500	1,260	1,710	1,230
b. Run								
1. Air or oxygen..... ^{2/}	1,890	604	-	-	-	190	130	190
.....cu.ft.per min. ^{2/}								
2. Steam..... lb. per hr.			1,200/2,100	1,150	1,200	1,860	1,020	-
Output:								
a. Blast gas.... cu.ft.per min. ^{2/}			1,800	2,990	2,580	1,550	2,270	1,570
b. Run gas.....do. ^{2/}	2,160	1,335	585	250	190	750	645	385
Properties of output gas:								
a. Blast								
1. Heating value..... ^{2/}			112	47	30	70	49	78
..... B.t.u.per cu.ft. ^{2/}								
b. Run								
1. Analysis..... percent:								
Carbon dioxide.....	9.7	47.8	20.8	35.8	40.8	20.0	27.1	16.7
Illuminants3	.3	.4	.6	.4	.4	.5	.4
Oxygen5	.2	.4	.3	.6	.2	.3	.3
Hydrogen	9.2	24.5	47.9	24.1	21.0	31.7	22.8	23.4
Carbon monoxide	13.1	21.2	12.0	8.9	6.9	10.6	9.4	9.8
Methane	1.5	4.1	7.6	7.5	7.2	3.3	3.5	4.2
Nitrogen	65.7	1.9	10.9	22.8	23.1	33.8	36.4	45.2
2. Heating value								
..... B.t.u. per cu.ft. ^{2/}	93	195	279	195	172	179	150	158
3. Specific gravity	0.94	1.00	0.62	0.92	0.98	0.78	0.90	0.84
4. Moisture content..... ^{2/}								
..... mol H ₂ O per mol dry gas ^{2/}	0.11	0.34	0.56	1.23	1.78	0.41	0.52	0.40
Overall coal utilization, basis of moisture- and ash-free coal								
Tons per day:								
a. Completely gasified	11.2	33.13 ^{3/}	6.4	8.5	6.5	8.9	9.7	7.2
b. Carbonized only ..	15.9	0	12.7	11.3	9.6	3.7	13.7	6.0
c. Total affected	27.1	33.13 ^{3/}	19.1	19.8	16.1	12.6	23.4	13.2
Overall heat balance ^{4/} , based on moisture- and ash-free coal affected plus steam introduced								
Percent:								
a. Heat of combustion of gases... produced.....	35.9	53.8	48.4	23.5	16.5	43.5	22.8	39.2
b. Heat of combustion of residual coke	38.3	0	42.9	37.8	39.0	19.0	38.7	29.7
c. Sensible and latent heat of moisture in gas	2.6	5.0	4.5	6.5	7.9	5.8	3.8	4.2
d. Sensible heat of dry gas	1.2	1.0	1.2	1.5	1.9	1.4	1.4	1.0
e. Unaccounted for.....	22.0	40.2	3.0	30.7	34.7	30.3	33.3	25.9

^{1/} Expressed as percent of the total moisture- and ash-free coal completely gasified during operation of the unit.

^{2/} Measured at 60° F., 30 inches Hg pressure, dry.

^{3/} Fresh coal plus coke from previous gasification with air, all calculated as equivalent coal.

^{4/} Temperature base, 60° F.

Examination of the composition of the product gases of table 6 indicates that it is possible to make gases of different types. The products from gasification with air might be utilized in generating power, while those from either oxygen gasification or water-gas operation could be used to produce synthetic liquid fuels.

The data for the three groups of water-gas cycles, given in columns 3, 4, and 5, show the effect of the age of a unit on both the quality and quantity of products. After a fraction of the coal is gasified and disappears from the reaction zone, poor contact occurs between the reactants, heat losses increase, and the operating temperature is reduced. These are some of the reasons for deterioration in quality and quantity of product with age of a unit.

In gasifying coal in place, it is convenient to consider the reaction as occurring in two stages: (1) Carbonization of the coal, with accompanying evolution of volatile matter, and (2) gasification of the coke remaining after carbonization. The extent of these two stages can be estimated by balancing the input against the output for a given system using carbon, hydrogen, and nitrogen balances. To employ these balances, however, it is necessary to know the composition and yield of the volatile carbonization products of coal and the coke residue. For many years, the Bureau of Mines has studied the carbonizing properties of coal, and the yield and the composition of the volatile products and the coke residues have been correlated with carbonizing temperatures. This correlation has been applied in estimating these items during underground gasification. For a specific coal, in this case America bed, it was possible to derive a series of three simultaneous equations for determining these values as follows:

$$(1) \quad X = 13 C - 9.7 H_2 + 19.4 O_2,$$

$$(2) \quad Y = 15.7 H_2 - .62 C - 31.4 O_2,$$

$$(3) \quad Z = .04 C - 3.54 H_2 + 43.1 O_2.$$

The symbols, based on 100 mols of dry product gas, are defined as follows:

X = pounds of coke gasified.

Y = pounds of volatile matter evolved.

Z = pounds of water entering reaction zone.

C = mol of carbon per 100 mols of dry gas. This carbon is derived completely from coal or adjacent carbon-bearing strata.

H₂ = total mols of hydrogen produced per 100 mols of dry gas. This hydrogen is derived from the coal and moisture flowing into the reaction zone. The hydrogen in the products is the sum of the hydrogen in the dry gas plus the hydrogen in the moisture accompanying the dry gas.

O₂ = total mols of oxygen per 100 mols of dry gas. The oxygen is derived from coal and water. The oxygen in the products is the sum of that appearing in the dry gas and that in the moisture accompanying the dry gas.

In table 6, the term "coal affected" represents the sum of the coal completely gasified plus that carbonized only.

The degree of utilization of the coal varies with the type of operation. In operations with air as the gasmaking fluid, more coal is carbonized than is gasified completely. This means that a quantity of coke remains underground when a unit has reached the end of its useful life. The ratio between coal gasified and coal remaining underground as coke has increased steadily as experience and improved control have been gained. Further improvements in technique should greatly reduce the amount of the coke residue indicated in column 1. When oxygen is used as the gasmaking fluid, no appreciable quantity of coke is left as a residue. It follows that increasing the oxygen content of air offers one method of eliminating most of the coke residue. In most instances during the steam runs and the mixed-air-steam runs, less residual coke was obtained than with air alone. The figures given represent the overall effect in which the blast period and run period are averaged. With the cycles used, the blast period overbalances the improvement realized during the run; nevertheless, exception for column 3, the results given in columns 4 to 8 inclusive are as good or better than for gasification with air alone (column 1).

On the basis of utilization of coal, the overall heat balances indicate that gasification with oxygen is the most efficient of the methods employed. The balance is based on total coal affected, and the improved efficiency reflects the influence of the complete gasification of all carbonaceous material.

Although the gases are relatively cool when they leave the ground in a well-operated system, the heat balances in table 6 show that the sensible heat content of the product gases is very low. This is because of the point chosen for measurement. A threefold increase in this value with a resultant decrease in the unaccounted-for item is the approximate magnitude of error introduced by choice of the measurement location.

The unaccounted-for heat represents chiefly heat losses to the strata surrounding the underground reaction zone. Part of this loss can be allocated to sensible heat in coke residues, but the rest probably is lost in heating and fusing surrounding rock strata and in vaporization of water which is recondensed in these strata. The heat allocated to residual coke should be included as the heat lost with the unaccounted-for heat. A decrease in underground heat losses should be a major objective of future experiments in underground gasification. Several methods may be effective in controlling these losses; increased production rates, simultaneous operation of several units located as near each other as possible, and control of water inflow into the reaction zone.

Heat balances in table 6 were reported on the basis of total coal affected. Heat balances recorded in previous reports were based on a carbon-balance calculation, wherein it was assumed that the carbon content of the products equaled the amount of coal gasified. The present method appears to be more useful although the calculation is sensitive to the cumulative errors of sampling, analysis, and measurement. If the calculations are made on a comparable basis, it will be shown that the thermal efficiencies for gasification with air have steadily improved during the experiments at Gorgas. Latest experiments show efficiencies half again to several times as great as those achieved during earlier trials.

SYNTHETIC FUELS DEMONSTRATION PLANT, LOUISIANA, MO.

The year 1953 brought to conclusion a significant phase of the Bureau of Mines synthetic liquid fuels program. Under authority of the Synthetic Liquid Fuels Act of 1944, the Bureau of Mines constructed and operated a coal-hydrogenation and gas-synthesis semiworks plant at Louisiana, Mo. The primary objectives were:

- (1) To demonstrate that American coals could be converted into liquid fuels, using American equipment and techniques.
- (2) To obtain and make available to industry the design and operating data necessary for determining the feasibility of converting coal into liquid fuels by high-pressure hydrogenation.

During 4 years of operations initiated in 1949 the above objectives were in general realized. Six American coals representing different ranks, from high-volatile bituminous to subbituminous and lignitic, were processed on a tonnage basis into synthetic motor-grade gasoline. The gasoline produced was laboratory and fleet tested and proved satisfactory as motor fuel. Engineering data were obtained from actual operation of the plants, which were carefully analyzed and studied by the Bureau and by private industry to determine the practicability of industrially converting coal into liquid fuels.

These studies indicated that the processes demonstrated in the Bureau's coal-to-oil plants were technically feasible but emphasized a need for evolution of economically practicable processes rather than continuance of the larger scale development work. As a consequence, operations at the Bureau's demonstration plants in Louisiana, Mo., were terminated in June 1953, and the Bureau's coal-to-oil Synthetic Liquid Fuels Program was oriented toward laboratory research and small pilot-plant process development.

Coal-Hydrogenation Demonstration Plant

Operations - Liquid Phase

The liquid-phase processing of a lignite was done in 1952 but too late in the year for inclusion in the report of that year's operations. Experimental runs were made to determine the efficiency and practicability of the following operations in removing solids from heavy oils:

- (1) Multiple centrifugation.
- (2) Carbonization.
- (3) Precoat filtration.

The liquid-phase run on lignite was preceded by a short run on Pittsburgh seam coal at mild hydrogenation conditions.

Liquid-Phase Run 10 (Pittsburgh-Seam Coal)

A 4-day run on Pittsburgh-seam coal was made to complete previous operations with this coal and to produce, under mild hydrogenation conditions, heavy oil for study by industry under cooperative agreement with the Bureau of Mines.

Pertinent operating conditions, yields, and analyses of coal and heavy oil products are presented in tables 7 and 8 under these mild hydrogenation conditions a good yield of heavy oil - 55 percent by weight based on m.a.f. (moisture- and ash-free) coal - was obtained. Liquefaction at 89 percent was lower than attained in the middle oil operations previously reported. Hydrogen consumption of 4 percent, based on m.a.f. coal, was higher than anticipated from similar German operations but consistent with the degree of conversion to middle oil and gasification.

TABLE 7. - Operational and yield data; heavy-oil operations, Pittsburgh-seam coal

<u>Operational</u>	
Pressure.....	p.s.i. 7,900
Conversion temp.	°F. 834
Coal input.....	m.a.f. tons/day 50.8
Pounds per cu.ft.hr.	23.5
Paste input.....	lb./cu.ft.hr. 66.0
Percent, m.a.f. coal.....	35.6
Paste oil.....	percent, middle oil 26.7
Catalyst addition, type.....	FeSO ₄ ·7H ₂ O
Percent on m.a.f. coal.....	2.0
<u>Yields.....percent, on m.a.f. coal:</u>	
Light oils.....	14.8
Soluble heavy oils (asphalt incl.).....	55.4
Asphalt make.....	28.5
Unliquified coal.....	11.0
Gasification (by diff. in carbon).....	12.0
<u>Reaction Hydrogen</u>	
Lb./lb. m.a.f. coal.....	0.04

TABLE 8. - Analyses of coal and heavy-oil products; heavy-oil operations, Pittsburgh-seam coal

Coal analyses, percent	M.f.	M.a.f.	As			
			rec'd.	M.a.f.		
Proximate:			Ultimate Analyses:			
Volatile matter	40.4	43.1	Moisture.....	2.6	-	
Fixed carbon...	53.4	56.9	Carbon.....	74.8	81.8	
Ash.....	6.2	-	Hydrogen.....	5.1	5.6	
Petrographic:			Nitrogen.....	1.4	1.5	
Anthraxylon.....	65		Sulfur.....	2.7	3.0	
Translucent attritus..	24		Oxygen.....	7.4	8.1	
Opaque attritus.....	7		Ash.....	6.0	-	
Fusain.....	4		C/H.....	14.7		
<u>Heavy-oil analyses</u>						
	Gravity, 60/60° F.	Insolubles, wt. %		Ash, %	Dist. at 620° F., %	
		Benz.	Pet. ether			
Light-oil bottoms	1.034	Nil	Nil	Nil	6	
H.O.L.D.	1.234	18.8	38.7	10.8	-	
Benzene-sol.	1.137	-	24.5	Nil	9	
<u>Ultimate composition</u>						
	C	H	N	S	O	C/H
Light-oil bottoms...	87.6	8.7	0.9	0.1	2.7	10.1
H.O.L.D.	77.9	6.6	1.2	2.9	.6	-
Benzene-sol.....	87.8	7.5	1.2	.2	3.3	11.7

Liquid-Phase Run 10 (Velva, N.D., Lignite)

Processing in the liquid phase of Velva (N. Dak.) lignite followed immediately the short run on Pittsburgh-seam coal. The run extended continuously from the latter part of October 1952 into December. This coal was similar to Lake DeSmet (Wyo.) coal processed during 1951. However, improvements for operations in cold weather had been made in the interim, and zero weather prevalent during processing of Velva lignite did not hamper operations seriously. Approximately 1,665 tons of Velva lignite was processed, yielding 120,000 gallons of vapor-phase charge stock and 30,000 gallons of heavy oils.

Description of Coal

Velva lignite is found in the Coteau bed in the extreme southeastern corner of Ward County, N. Dak. The bed is 10 to 18 feet thick and comprises an area of some 250 square miles underlying parts of 8 or 9 townships. The coal processed was mined about 10 miles southwest of Velva, N. Dak.

Analyses of coal are given in table 9. The high ash and alkalinity are objectionable characteristics of a coal for hydrogenation, requiring neutralization for normal processing. The low carbon content and high percentage of organic materials relatively inert to hydrogenation are conducive to a low yield of oil. The high moisture inherent in this type of coals requires extra capacity and greater fuel consumption in the grinding and drying operations. However, despite these objectionable characteristics, the abundance and availability at low cost of such lignites justify development work with them.

TABLE 9. - Coal analyses; Velva (N. Dak.) lignite

<u>Proximate, percent:</u>	<u>As</u>		<u>Ultimate percent:</u>	<u>M.f.</u>	<u>M.a.f.</u>
	<u>rec'd.</u>	<u>M.a.f.</u>			
Moisture.....	35.9	-	Carbon.....	63.0	70.6
Vol. matter.....	28.3	49.5	Hydrogen.....	4.2	4.7
Fixed carbon.....	28.9	50.5	Nitrogen.....	1.1	1.2
Ash.....	6.9	-	Sulfur.....	.7	.7
			Oxygen.....	20.2	22.8
<u>Petrographic, percent:</u>			Ash.....	10.8	-
Anthraxylon.....		61	C/H.....	15.0	
Trans. attritus..		27			
Opaque attritus..		9	<u>Alkalinity:</u>		
Fusain.....		3	H ₂ SO ₄ /kg. coal..		
		grams		70.5

Processing

Operating conditions and yields typical of the liquid-phase operations on Velva lignite are given in table 10 and the analyses of process and product streams in table 11.

TABLE 10. - Operational and yield data; liquid-phase processing of Velva lignite

	Period	Period
	I	II
Operational:		
Pressure.....p.s.i.:		
Inlet.....	8,400	8,400
Outlet.....	7,600	7,800
Conversion temperature.....°F. (average)	858	846
Coal, moisture - free.....tons/day	58.9	50.8
Coal, moisture and ash free.....do.	51.8	44.8
Coal, moisture and ash free.....lb./cu.ft.hr.	24.0	20.7
Paste.....do.	75.6	75.0
Paste.....percent m.a.f. coal	31.7	27.6
Paste oil.....percent <620° F.	13.7	12.2
Total gas to converters..cu.ft./lb.m.a.f. coal	85.2	106.7
H ₂percent by volume	83.4	81.9
Make-up H ₂cu.ft./lb.m.a.f. coal	18.8	21.8
Catalyst.....percent on m.f. coal:		
FeSO ₄ ·7H ₂ O.....	3.1	1.6
(NH ₄) ₂ MoO ₄	-	.4
Yields.....wt. percent on m.a.f. coal:		
Total gases.....	18.9	18.1
Hydrocarbon gases.....	11.9	11.1
Light oils, C ₅ + to 620° F.	47.4	49.9
Benzene-soluble heavy oils, >620° F.	15.0	13.5
Asphalts.....	3.0	4.2
Coal liquefaction.....	94.6	94.7
Oil yields.....bbl./ton m.a.f. coal:		
L.P. gases.....	0.6	0.5
Light oils.....	2.9	3.1
Heavy oils (benzene-soluble).....	.8	.7
Total oils.....	4.3	4.3
Space time yields.....lb./cu.ft.hr.		
Light oils.....	11.4	10.3
Heavy oils.....	3.6	2.9
Hydrogen consumption.....lb./lb. m.a.f. coal		
Total H ₂	0.094	0.109
Reaction H ₂058	.059

TABLE 11. - Typical analyses of process streams and products; liquid-phase hydrogenation of Velva lignite

	Catalyzed m.f. coal	Paste oil	H.O.L.D.	L.O.B.	Middle oil	Naptha	Gasoline
Ultimate...wt. percent							
Ash.....	12.2	6.4	16.9	Nil	-	-	-
Carbon.....	61.8	82.7	73.4	88.2	86.7	82.9	84.3
Hydrogen.....	4.3	7.5	6.1	8.4	9.4	9.8	13.1
Nitrogen.....	.9	.9	.8	.8	.9	.8	.3
Sulfur.....	.8	.5	1.3	.03	.05	.04	.01
Oxygen.....	20.0	2.0	1.5	2.6	2.9	6.5	2.3
C/H.....	14.3	11.0	12.0	10.5	9.2	8.5	6.4