TABLE 9. - Comparison of contents of various components in 50° - 150° C. cuts of liquid- and vapor-phase gasoline from Rock Springs coal

Components in 50° - 150° cuts	Pounds per ton of coal		
	Liquid phase	Vapor phase	Vapor-phase cat. aromatized 1/
Paraffins, total	27.6	143.5	98.8
Naphthenes, total	71.4	384.0	91.2
Cyclopentane	0.3	2.6	2:1
Methylcyclopentane	3.6	64.6	37.9
Cyclohexane	10.8	26.6	5.1
Dimethylcyclopentanes	4.5	53.2	22.9
Methylcyclohexane	12.7	52.7	6.8
Ethylcyclopentane	4.8	38.9	4.4
C_8 - Naphthenes)	17.5	78.3	12.9
C_9 - Naphthenes)	10.5	67.2	
Aromatic, total	33.5	208.7	412.0
Benzene	2.5	46.1	86.8
Toluene	11.0	83.3	158.3
Ethylbenzene	11.6	23.7	47.4
m-p-Xylene	5.3	46.8	95.8
o-Xylene	2.9	8.8	22.9
Olefins, total	15.2	8.3	13.7
Total	147.7	744.5	614.9
Nonhydrocarbons, total	11.3	10.6	

1/ Probable yields had the vapor-phase cut been subjected to catalytic aromatization. Estimates based by the Bureau on results obtained by the Standard Oil Co. of Indiana upon hydroforming of vapor-phase gasoline through a cooperative agreement. Full results of cooperative work will be published later.

Gas-Synthesis Demonstration Plant

Coal Gasification

Installation of the new Morgantown-type vertical oxygen-coal gasifier (fig. 14), described in the 1950 Annual Report, has been completed. A flow diagram of this system, which employs a new method of feeding preheated steam-coal mixtures, is shown as figure 15. Existing equipment was used as far as possible in the hook-up.

The coal-feeding system consists of accumulator metering tanks for coal and water, which dump periodically into a slurry-mix tank as additional material is required in the latter. This tank is equipped with two high-speed, propeller-type agitators and is of 1,600 gallons capacity, large enough to

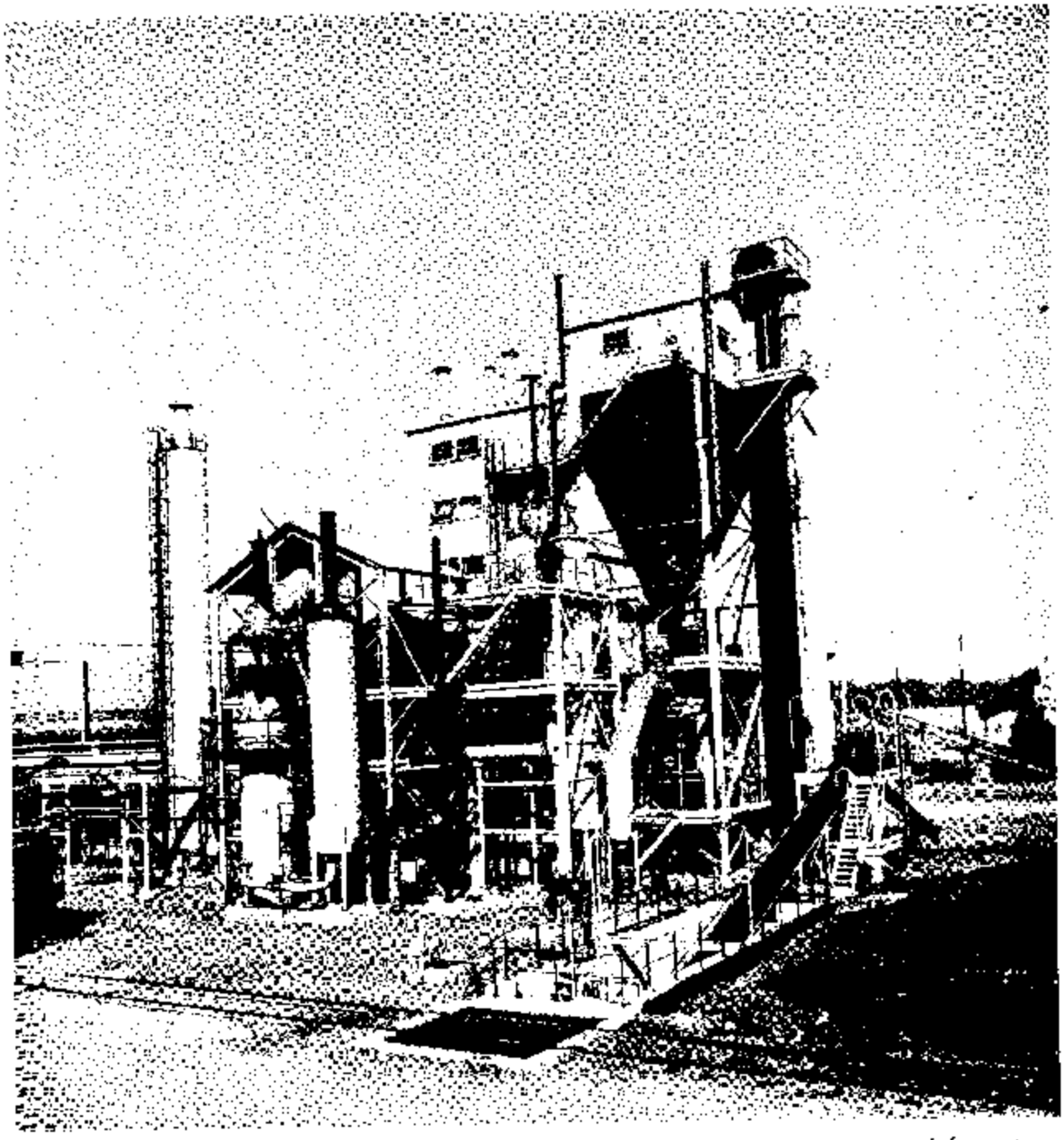

Figure 14. - Coal-Gasification Unit. On right is Kerpely coke gasification unit, on left next to tall washer-cooler is Morgantown-type vertical gasifier and steam superheater.

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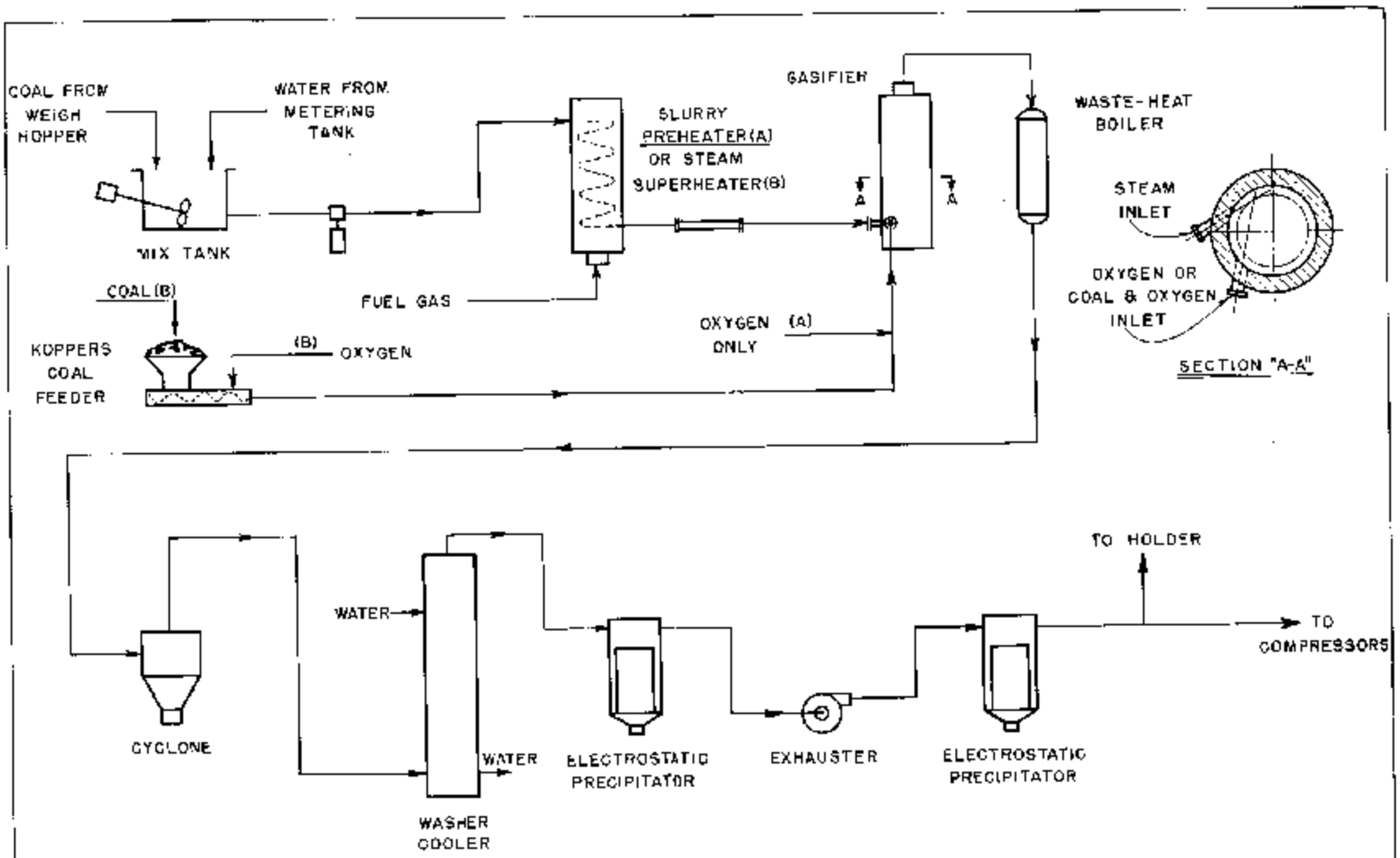

Figure 15. - Schematic flow diagram - gasification.

provide several hours' operation and minimize any changes in concentration owing to intermittent dumping. The discharge line from this tank is about 6 inches above the bottom of the tank and is connected to the suction of a reciprocating pump, motor-driven through a variable-speed oil gear. The pump discharge enters the top of a fired coil in which the water is vaporized, and the resulting steam-coal mixture is superheated to 900° to 1,000°F. The coil consists of a preheater section of several turns of 3/4-inch pipe, followed by an evaporating section of 2-inch pipe, which, in turn, leads into the superheating section, made of 3-inch, 4-6 chrome tubing. The transfer line from the coil discharge to the gasifier was kept as short as possible and is heavily insulated. The heated steam-coal mixture was introduced into the gasifier about 1-1/2 feet above the bottom and tangent to a circle of slightly smaller diameter than the gasifier itself. Oxygen is brought in through another nozzle at the same level and tangent to the same inner circle. The gasifier proper consists of a vertical refractory-lined cylinder, 4 feet, 4 inches, inside diameter and approximately 20 feet in height. Provision was made for measuring temperatures and pressures throughout the length of the unit. The refractory is high-purity aluminum oxide that was ram-packed against inside forms and fired in place to produce a monolithic lining. This material has the advantage of higher melting point and much higher resistance to thermal shock than the silica brick used in the previous unit. If tests here show that the monolithic lining is satisfactory, important economies in construction costs may result. Under a cooperative agreement, the Aluminum Co. of America supplied much valuable technical assistance and the major part of the material.

Products from the gasifier pass out the top and into the same waste-heat boiler and gas-handling system that were used with the previous gasifier - that is, a cyclone dust catcher, gas-washer cooler, electrostatic dust precipitators, and gas exhauster.

To test operation of the coal slurry-feed system independent of the gasifier operation, the connection between the slurry preheater and gasifier was removed so that the material leaving the preheater coil would discharge into the air and its characteristics could be observed. On the first tests, there was some trouble with plugging of the coil, and the discharge stream was quite erratic, alternately blowing almost clear steam and very dense clouds of coal dust. An orifice then was installed in the discharge line to hold a back pressure of about 15 pounds on the coil, and the uniformity of discharge seemed definitely improved but still unsatisfactory. A smaller orifice was installed, which gave a back pressure of about 40 pounds, and a further improvement was noted, although it was felt that the stream still was not uniform enough to warrant use as a gasifier feed. Further increases in pressure could not be made on this coil without exceeding its safe working pressure at the temperatures encountered.

Pumping difficulties occasionally had been encountered and in every instance a small amount of very coarse coal particles was found under the pump valves. To eliminate this, a vibrating "scalper" screen was installed in the coal-pulverizing system to remove the +30-mesh material.

At this point the necessity for determining the operability of the gasifier proper led to the decision to postpone further work on the slurry feed and adapt one set of Koppers screw feeders to use on this new unit. The slurry feed heater was used as a steam generator and superheater.

The slurry method of feeding, if successful, would have its greatest advantage in connection with a gasification unit operating under elevated pressures. Plans are now in progress for a new coil designed to operate at appreciably higher discharge pressures. If this higher pressure makes the operation satisfactory, the unit as constructed will be suitable for use when a demonstration-scale pressure-gasification unit is installed. Until that time it can be used as feed for a gasifier operating at atmospheric pressure.

Tests on New Gasification Unit

Several runs have been made in the vertical gasifier, and the results of four were reliable enough to warrant calculation. Best results were achieved in a 3-day run with the unit on stream about 70 percent of the time. Owing to difficulty with the coal feed screws, the oxygen:coal ratio was above that planned, and ranged between 11 and 12 cubic feet of oxygen per pound. Carbon conversion was 97 to 99.5 percent. All of the stable operating periods and the average of the whole run were appreciably better than any results obtained on the previous unit, and the results of the best period of operation closely approach those achieved in the Morgantown vertical pilot-plant unit on this same coal.

During this run there was evidence that deposition of slag in front of the burner nozzle deflected the oxygen-coal flame so that it impinged directly on the lining. As a result of local high temperatures, a considerable portion of the alumina was melted out, and the run was ended with 12 to 15 inches of an extremely hard, high-melting mixture of aluminum oxide and coal slag in the bottom of the gasifier.

After this run, it was felt that a change in the oxygen-coal nozzle to make it tangent to a smaller circle would help prevent the build-up of slag deposits from the wall to the point where it could block the oxygen-coal stream. Examination of the gasifier after the run indicated that the main path of the superheated steam was below that of the oxygen-coal stream within the gasifier and that better mixing would be achieved if the steam were directed slightly upward. A new steam nozzle was installed to achieve this. The crater from which the alumina had been melted was patched with small, prefired alumina shapes, cemented in place by an alumina ram mix.

Two runs were made after these changes; the results were definitely inferior to those of the preceding run and little if any superior to those achieved on the Koppers gasifier. Inspection of the unit after these runs showed no evidence of damage to the refractory, either from melting or erosion. Present efforts are directed toward further changes that will protect against damage to the refractory and still permit the efficient operation previously achieved.

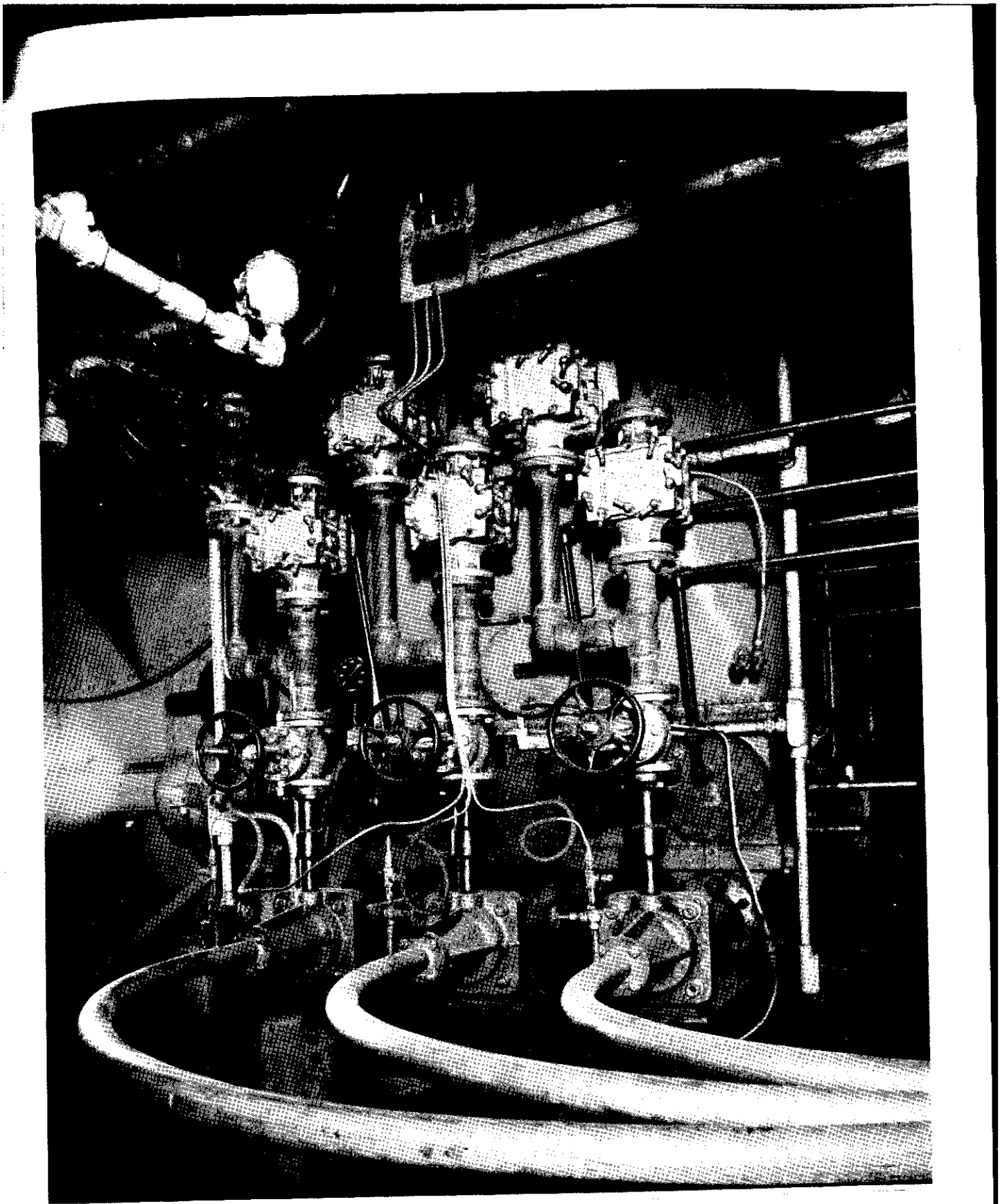

Figure 16. - Pulverized-coal screw feeders and coal-transfer tubes in coal-gasification unit.

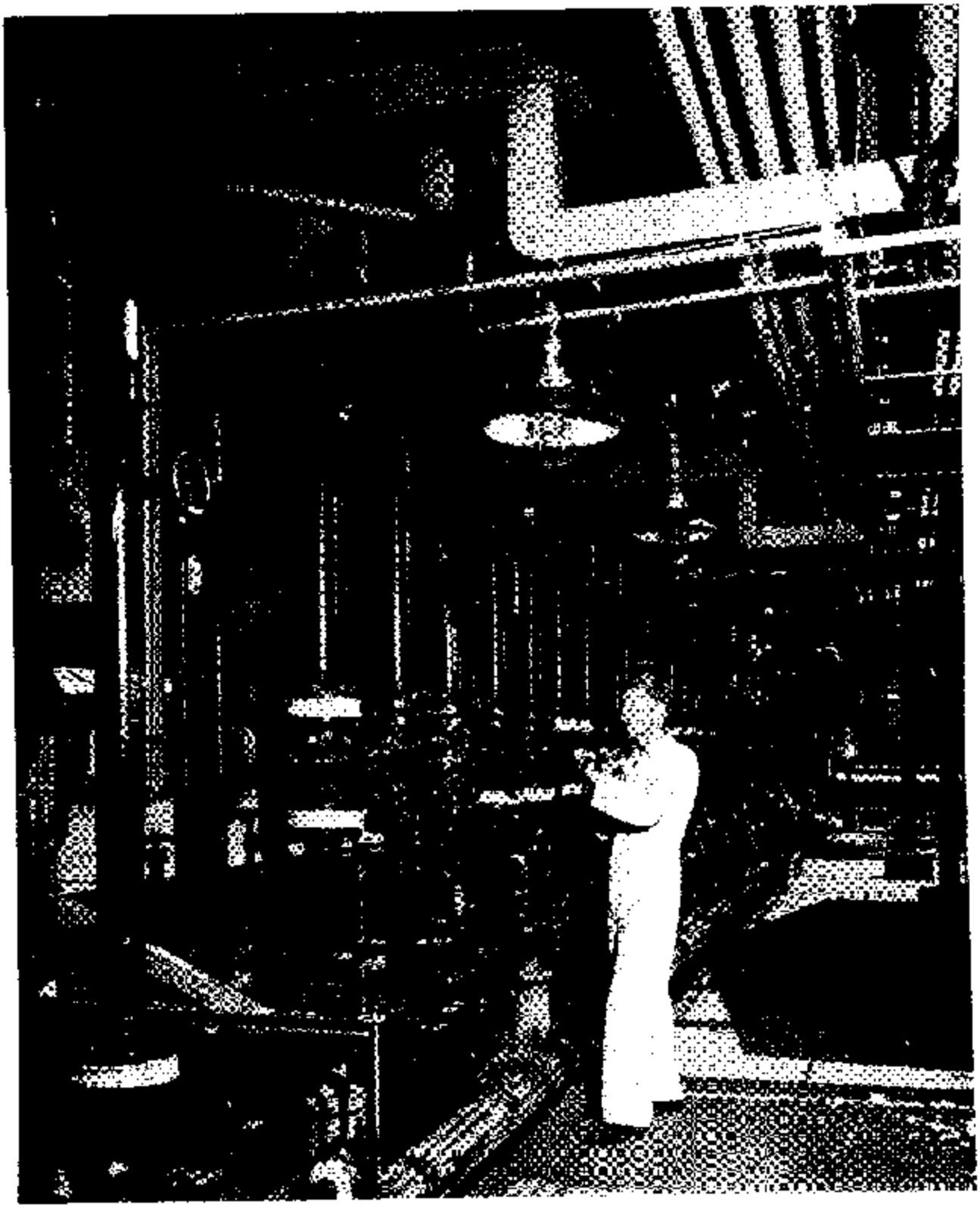

Figure 17. - Piping and manifolding in gas-purification unit that removes sulfide from synthesis-gas stream.

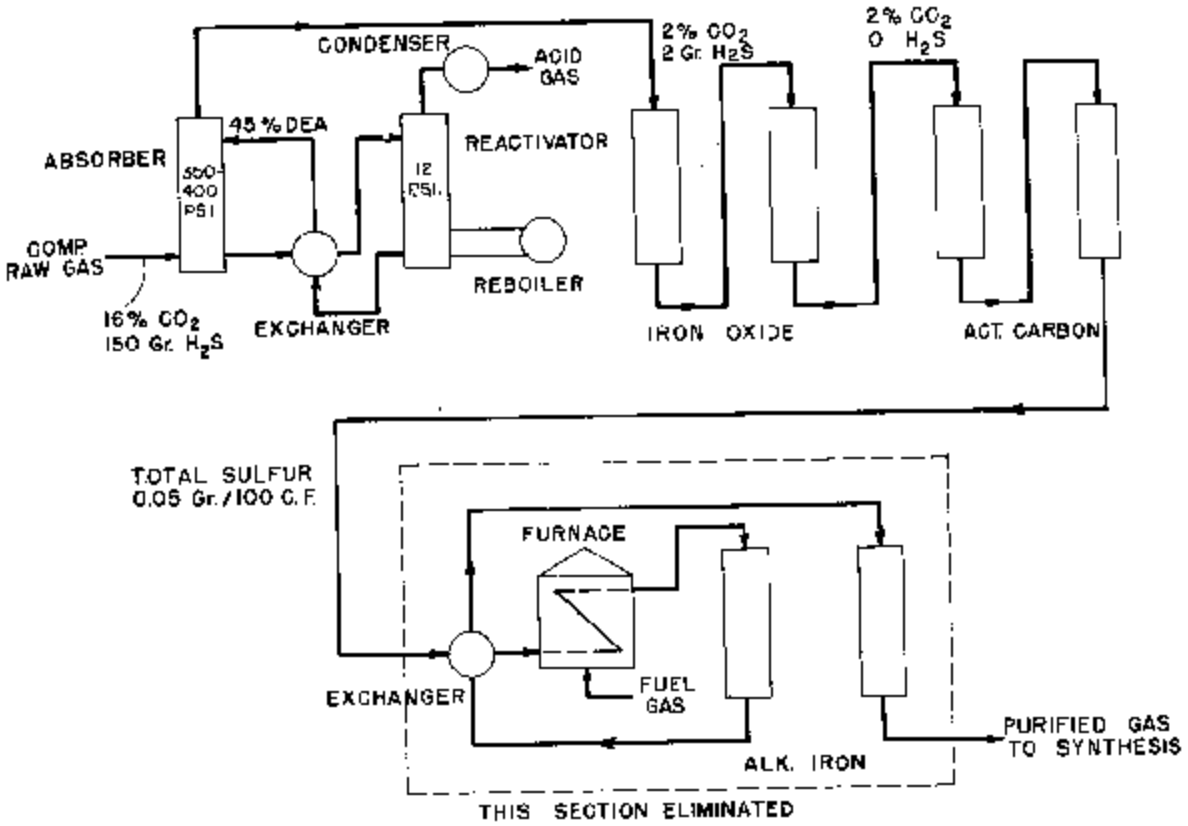

Figure 18. - Schematic flow diagram - synthesis-gas purification.

Further work on this unit was postponed to test and put into operation the gas-purification section of the plant, in preparation for the forthcoming synthesis run. In testing the purification unit, the Kerpely producer, using oxygen, coke, and steam, was employed as a source of synthesis gas and was operated for several extended periods. Between May and November 1951, the producer was in operation for a total of about 1,400 hours. During this time 23,000 M standard cubic feet of process oxygen and 1,450 tons of coke were fed, producing 113,000 thousand standard cubic feet (M std. cu. ft.) of make gas. The gas produced normally is of the following composition:

CO_2	percent	16
H_2	do.	36
CO	do.	46
H_2S	grains per 100 cu. ft.	150

On an over-all basis, these production figures indicate a requirement of 30.8 pounds of coke as charged and 243 standard cubic feet of oxygen (100 percent) per thousand cubic feet of CO + H_2. This compares very favorably with the published reports of commercial oxygen-blown producers.

In the operations during September and October 1951, about 45 million cubic feet of gas was produced and about 40 million cubic feet purified. A substantial part of the purified gas produced during these later operating periods was used as feed for the Fischer-Tropsch synthesis unit.

Operation of the producer and its auxiliaries, although not entirely trouble-free, has been quite steady and reliable. Some trouble was encountered with deposits of very heavy tar in the gas exhauster. As originally installed, there were two electrostatic precipitators in series between the exhauster and the gas compressors to remove dust from the compressor feed gas. Piping was changed to put one of these precipitators in the gas line leading to the exhauster. This almost completely eliminated the tar deposits in the exhauster, and the single precipitator in the compressor feed line was adequate to remove dust and tar.

Operation of Purification Unit

The problem of purifying synthesis gas is one that has been given a great deal of study. The tolerance of the Fischer-Tropsch catalysts for sulfur compounds is extremely low, and it had been hoped to produce a gas containing not more than 0.1 grain of sulfur per 100 cubic feet. Later, bench-scale and pilot-plant studies on the synthesis indicated that carbon dioxide also should be as low as possible. A photograph and a flow diagram of the purification system as originally installed are shown in figures 17 and 18. The cleaned gas is compressed to synthesis operating pressure and scrubbed to remove carbon dioxide and hydrogen sulfide with a 30- to 45-percent solution of diethanolamine in water. Used diethanolamine is expanded to about 12 pounds pressure and sent to a reactivator wherein the combined action of decreased pressure, heat, and water vapor removes the absorbed carbon dioxide and hydrogen sulfide, which then are vented to the flare stack. The revivified diethanolamine is cooled and pumped back to the absorber. Synthesis gas leaving the absorber contains only about 2 percent of carbon dioxide and 1 to 2 grains of hydrogen

sulfide per 100 cubic feet. It is passed downward in series through two beds of iron oxide on wood shavings, which remove completely the residual hydrogen sulfide. To remove the organic sulfur compounds, the gas next is passed downward through two beds in series filled with active carbon. This material has the property of adsorbing and retaining the organic sulfur compounds. When the carbon in one bed becomes saturated, it is removed from the line and regenerated by blowing with superheated steam. Then it is returned to service as the second bed.

Almost without exception, in German synthetic-fuel operations, beds of hot iron oxide-soda ash mixture were employed as a final purification measure. This material decomposed the organic sulfur compounds which passed the active carbon and absorbed the hydrogen sulfide formed. As a precaution, similar beds were included in the design of the Louisiana purification system. It should be noted, however, that the German purification plants operated under substantially atmospheric pressure. In the laboratory-scale purification work at Morgantown, W. Va., it was found that the active carbon would remove organic sulfur completely when operated 300 to 400 p.s.i., so that it was generally felt that the iron oxide-soda ash mixtures were not necessary. Also, economic calculations had shown that removal of organic sulfur by active carbon was very much cheaper than by the German system.

Mechanical operation of the purification unit was tested, and the preliminary operator training was accomplished by "dummy" runs in which inert gas was used instead of synthesis gas. After a short period of such operation, the purification unit was first put into service from May 29 to June 8, 1951, on gas made in the Kerpely producer. There were, of course, many operating difficulties and disappointments, but even in this first period it was rather clearly demonstrated that the gas ultimately could be purified to the degree required for the synthesis unit. Since this first trial, there have been four major periods of operation, each more reliable and more satisfactory than the preceding one. During one of the early operations, considerable trouble was experienced with the iron oxide-soda ash mixtures. It was found that, during operation under these elevated pressures, particularly with traces of oxygen present in the gas, undesirable side reactions were promoted that led to release of heat and very difficult temperature control. In one instance this resulted in overheating until the vessel wall failed. In view of the substantially complete removal of organic sulfur by active carbon under the operating conditions, it was decided that the iron oxide-soda ash mixture was unnecessary and undesirable, and the piping has been changed to eliminate it from the system.

In the last two periods 40,000 thousand cubic feet of gas was processed, during a total operating time of 540 hours. In present operations, the feed gas to the synthesis unit consistently contains less than 2 percent carbon dioxide, and the total sulfur content in general is well below 0.05 grain per 100 cubic feet.

As the gas from the Kerpely producer is similar in composition and character of impurities, it is felt that the present operation gives full assurance that the gas from any high-temperature coal-gasification unit can be purified

to the extent required for the Fischer-Tropsch synthesis. Work is to be continued on this unit to improve the operating efficiency and reduce the cost of the purification step.

Operation of Oxygen Plant

The oxygen plant continued in operation as required for the coal-gasification experiments and later for the Kerpely producer. To November 1, the plant had operated 3,000 hours during the year and produced 60 million cubic feet of product oxygen. Only twice have there been unplanned interruptions to the oxygen-plant operation. In one, a German motor on the low-pressure compressor failed in the windings. A new motor was procured, and the German motor was rewound to serve as a spare. Later a bearing on the new motor overheated badly, and the rewound German motor was reinstalled, with a total interruption to oxygen production of only 10 hours. During this time the purification and synthesis plants operated with reduced throughput from the storage holder, so that no shutdown was necessary.

Theoretical Studies

The thermodynamic studies of coal gasification mentioned in previous reports have been extended to cover substantially all probable operating conditions. These studies determine what theoretically may be obtained from a given coal under specific conditions but do not give information as to the effect of time, temperature, and pressure on the relative speed and degree of completion of the various reactions involved. Because of the complex nature of coal, the high temperatures involved, and the important effect of heat losses on results, basic laboratory research on this problem is extremely difficult.

It was felt that a mathematical attack on this problem, using what fundamental information is available, and later comparison of calculated with actual operating results might be a more feasible approach.

After a study of published information, the basic assumptions were formulated and work started on a method of calculation. Each small-time increment involves a change in the temperatures, composition of the reaction mixture, the particle size of the coal, and many other factors. Because of the many variables and the complexity of the calculations, it was found that about 64,000 computations are required to solve a single case involving a single set of operating conditions.

Fortunately the equations could be cast in a form that would make possible the use of the International Business Machine (I.B.M.) sequence calculator used by the Explosives and Physical Sciences Division at Pittsburgh. This machine will make the 64,000 calculations in 6 hours' operating time. The actual computation work was begun near the end of the year. Twenty cases will be calculated, covering variations in oxygen:coal and steam:coal ratios at various temperatures.

After this work is completed, it should be possible to study the theoretical effect of these changes upon the progress of the various reactions involved. Comparison of these results with those of actual operation of the gasifier at Louisiana and others then should lead to a better understanding of the real effect of these variables and point the way to the most efficient operation.