Heat-balance calculations showed that the thermal efficiency of the process (100 times ratio of B.t.u. in gas plus residue to B.t.u. in coal) was 74 to 82 percent, notwithstanding the high heat losses encountered due to the comparatively low throughput. The temperature of the generator during the runs was 2,200° to 2,450° F. on the top and 1,250° to 1,750° F. at the outlet on the bottom, depending on the combination of experimental variables. Under these conditions the total steam decomposed was always at least 50 percent.

Synthesis-Gas Treating and Testing

Analytical Work

The Gas Treating and Testing Laboratory carried out the necessary analytical work for the experimental work on pulverized-coal gasification as well as underground gasification. Service aid in the instrumentation for the station also was performed.

Major effort continued to be devoted to the improving or developing of analytical methods required in gas purification. Work continued on the method for determining thiophene sulfur. With this method, thiophene is scrubbed from the gas with sulfuric acid, and as little as 0.0001 grain of thiophene sulfur can be determined in approximately 50 milliliters of acid.

Studies of the determination of very low concentrations of organic sulfur in gas were continued. These studies included a method whereby organic sulfur is converted to hydrogen sulfide by means of a hot platinum spiral and the resulting hydrogen sulfide is determined by the ultra-sensitive methylene-blue test, as developed by this laboratory.

Further studies were made of methods for determining organic sulfur in gas by combustion followed by turbidimetric determination of barium sulfate. This work included application of the spectrophotometer for turbidimetric measurement.

Existing methods for sampling and determination of dust in gas were studied to learn the effect of various variables on their reliability (fig. 63).

Bench-Scale Purification Experiments

Small-scale experiments were made to determine the efficacy of various methods for gas purification. These included work with catalysts for the removal or conversion of organic sulfur (fig. 64), with active carbons for removal of thiophene and gum-forming constituents, and scrubbing solutions for hydrogen sulfide removal. A preliminary study of the pyrolysis of thiophene was undertaken to determine the extent of such decomposition at temperatures that may prevail in the pulverized coal gasification process.

One commercial catalyst was investigated which, while not giving sufficiently complete conversion of organic sulfur, is considered worthy of further study. This catalyst is capable of promoting the conversion of organic sulfur to hydrogen sulfide in the presence of high concentrations of hydrogen sulfide. In pure hydrogen, to which organic sulfur and hydrogen sulfide have been added, conversion is substantially complete, leaving less than 0.1 grain of organic sulfur unconverted per hundred cubic feet of gas. With synthesis gas containing carbon monoxide, however, approximately 2 grains of organic sulfur (carbonyl sulfide) per hundred cubic feet of gas remains unconverted. The catalyst manufacturer is making further studies of the catalyst to improve its performance.

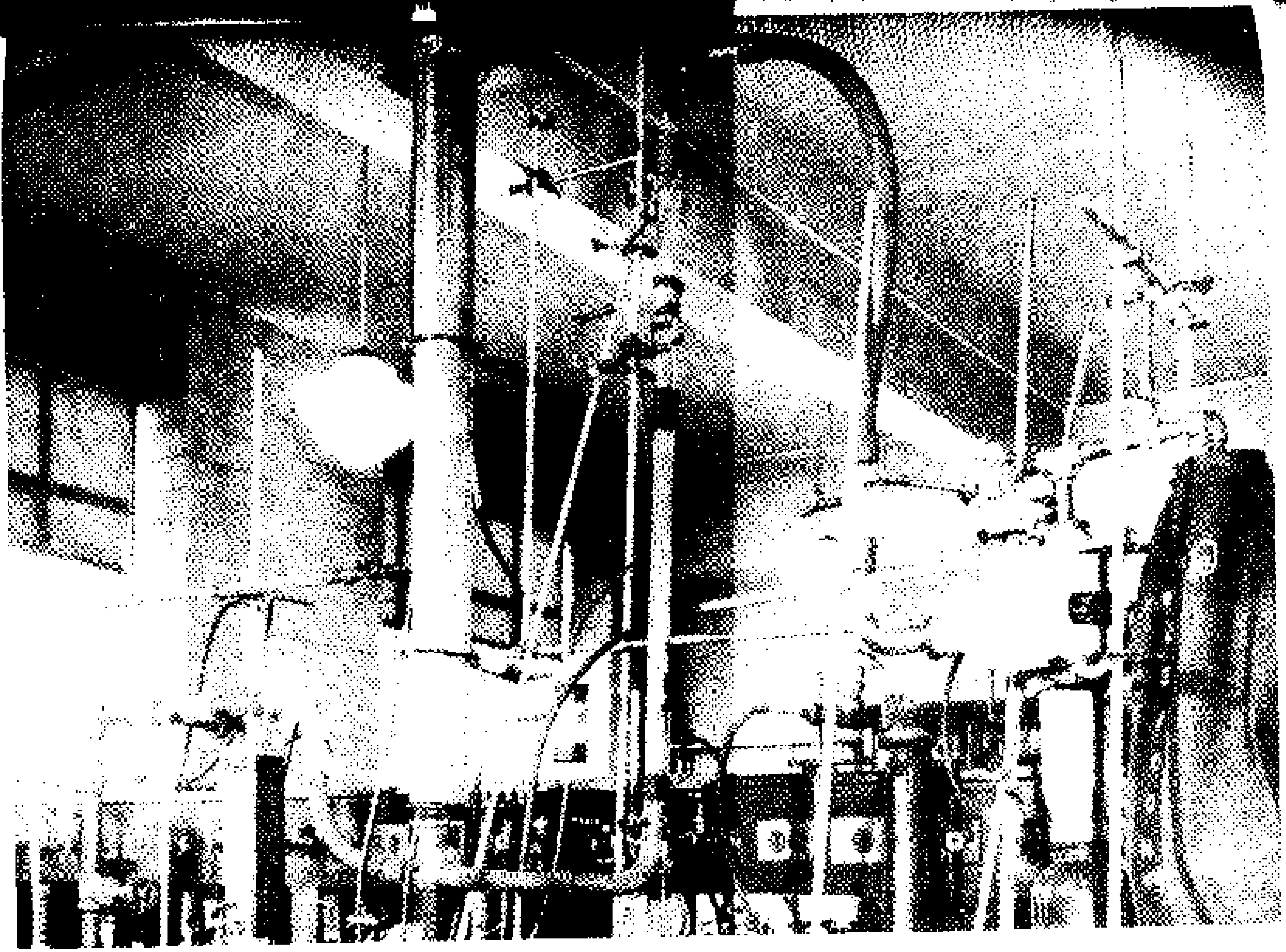

Figure 63. - Apparatus used in studying methods for sampling dust in gas.

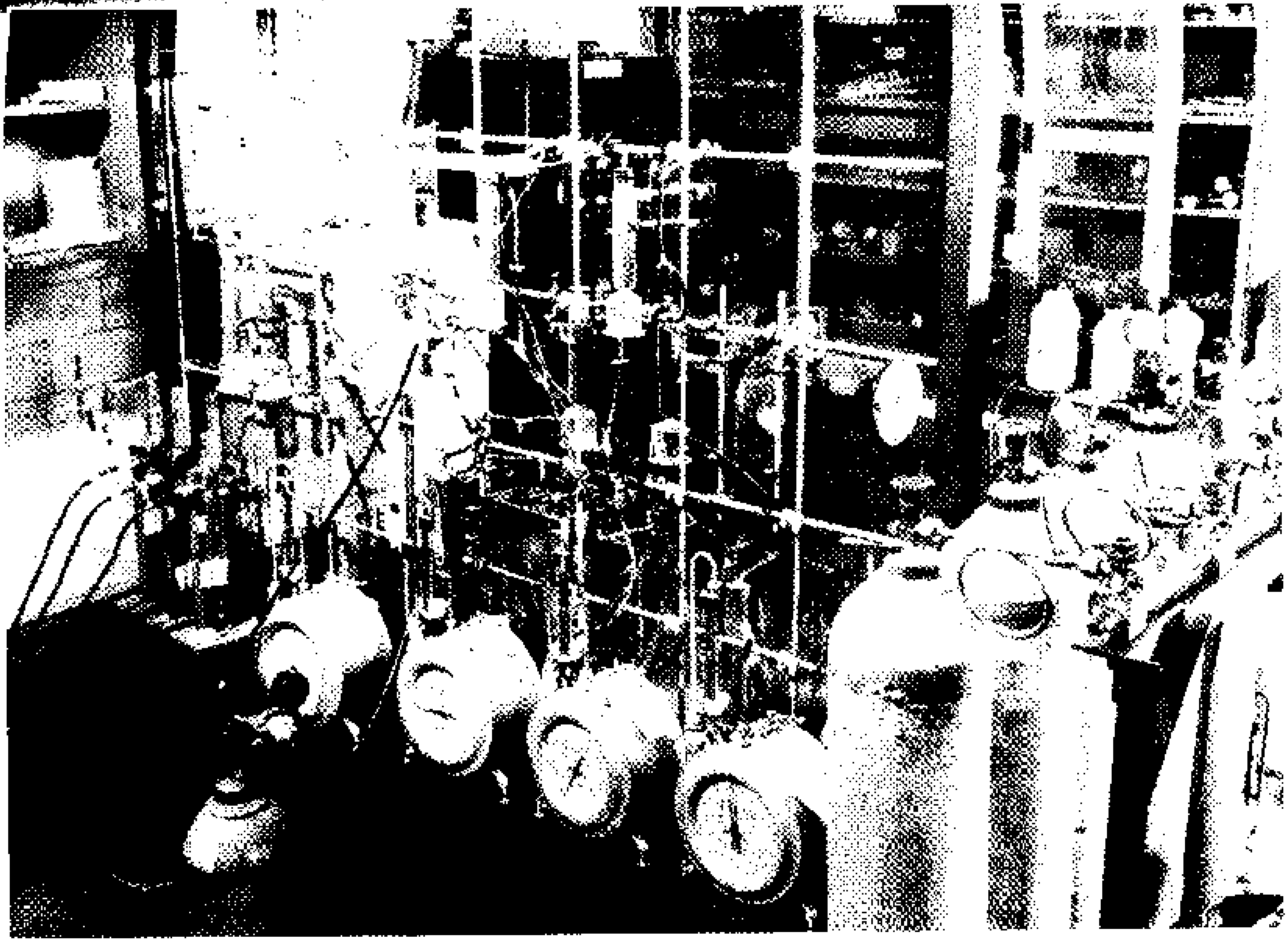

Figure 64. - Apparatus for removing organic sulfur from gas.

Incomplete studies of a cobalt molybdate catalyst gave encouraging results for the conversion of all forms of organic sulfur (including thiophene), but this catalyst was not successful in the presence of high concentrations of hydrogen sulfide.

In the absence of an initial high concentration of hydrogen sulfide, this catalyst was successful in reducing 35 grains of organic sulfur (9 grains as thiophene, 16 grains as ethyl mercaptan, 10 grains as carbon disulfide) down to 0.69 grain per hundred cubic feet. Further investigations will be made with this catalyst in an attempt to improve the removal by varying operating conditions.

Studies in progress with catalysts capable of removing both organic sulfur and hydrogen sulfide simultaneously are giving promising results. The catalysts absorb the sulfur and can be revivified by burning off the sulfur with air. Preliminary results show that gas containing 260 grains of hydrogen sulfide and 18 grains of organic sulfur (carbon disulfide and ethyl mercaptan) was purified to such an extent that the exit gas contained only 1.8 grains of hydrogen sulfide and 0.39 grain of organic sulfur.

These catalysts are of considerable interest because, if successful, they would make possible elimination of the liquid-purification step for removing hydrogen sulfide, leaving only small amounts of hydrogen sulfide and organic sulfur to be removed by final purification, thus appreciably reducing costs.

Investigations of active carbon for removal of thiophene and gum-forming hydrocarbons have been carried out (fig. 65). Carbons have been tested which show that the gum formers can be adequately removed but with considerable loss of carbon life in each revivification. Carbons examined for thiophene removal have shown considerable promise at ordinary pressures. With gas containing 4 grains of sulfur, as thiophene, complete removal was effected for 20 to 25 days, at a space velocity of 400, even after several revivifications with steam. Results with American active carbons were found to be superior to those obtained from "supersorbon," which was reputed to be the best of the active carbons used in the German synthetic-liquid-fuels industry. Work with active carbon will be continued at pressures of about 20 atmospheres.

Laboratory vapor-pressure determinations were made on various solutions used for removing hydrogen sulfide, including mono-ethanolamine, tri-ethanolamine, and alkazid "dik" solution. The mono-ethanolamine solution (15 percent) and the alkazid solution (sp. gr., 1.11) showed approximately the same carrying power. With a sulfur content of 2,000 grains of sulfur per gallon of solution, the hydrogen sulfide vapor pressure was 500 grains of sulfur per 100 cubic feet. Data were obtained on 20- and 50-percent tri-ethanolamine solutions having sulfur contents as high as 2,000 grains per gallon, resulting in vapor pressures ranging from zero to 18,000 grains of sulfur per 100 cubic feet. Tests were conducted to study the effect of carbon oxysulfide and carbon disulfide on tri-ethanolamine solutions. Neither proved detrimental.

Purification-Cost Study

In collaboration with the Gasification Planning Section of the Louisiana, Mo., Demonstration Plant, a cost study was made to estimate the cost of gas purification when coals of varying sulfur contents are used for gasification. The costs arrived at should be considered tentative only, for further work in the laboratory, gas purification pilot plant, and demonstration plant will make possible more accurate estimations.

Table 16 summarizes the gas-purification cost estimates for coals containing 1, 3, and 5 percent sulfur in a plant producing 10,000 barrels of liquid fuel per day.

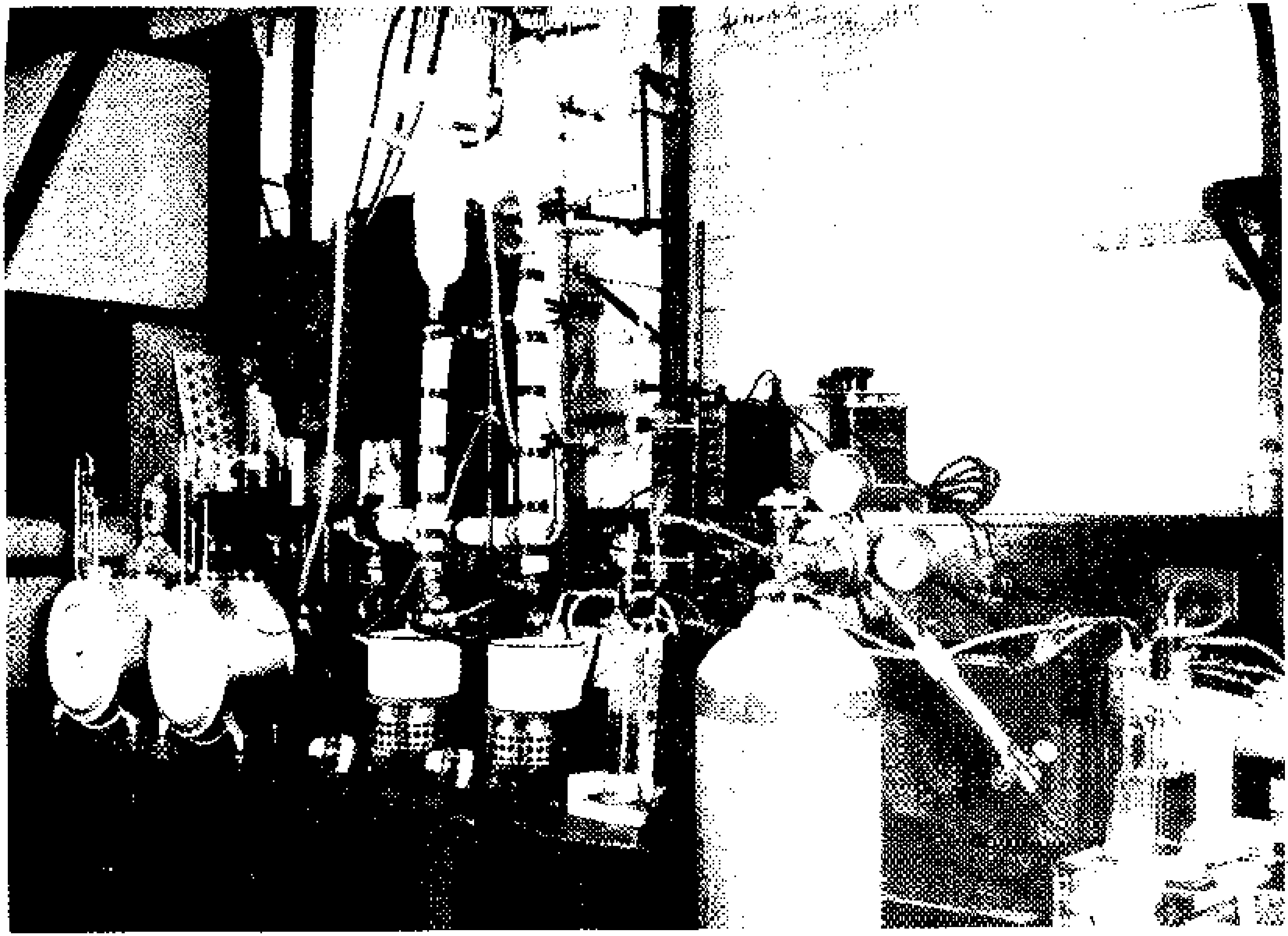

Figure 65. - Apparatus for evaluating activated carbons used in removing gum formers.

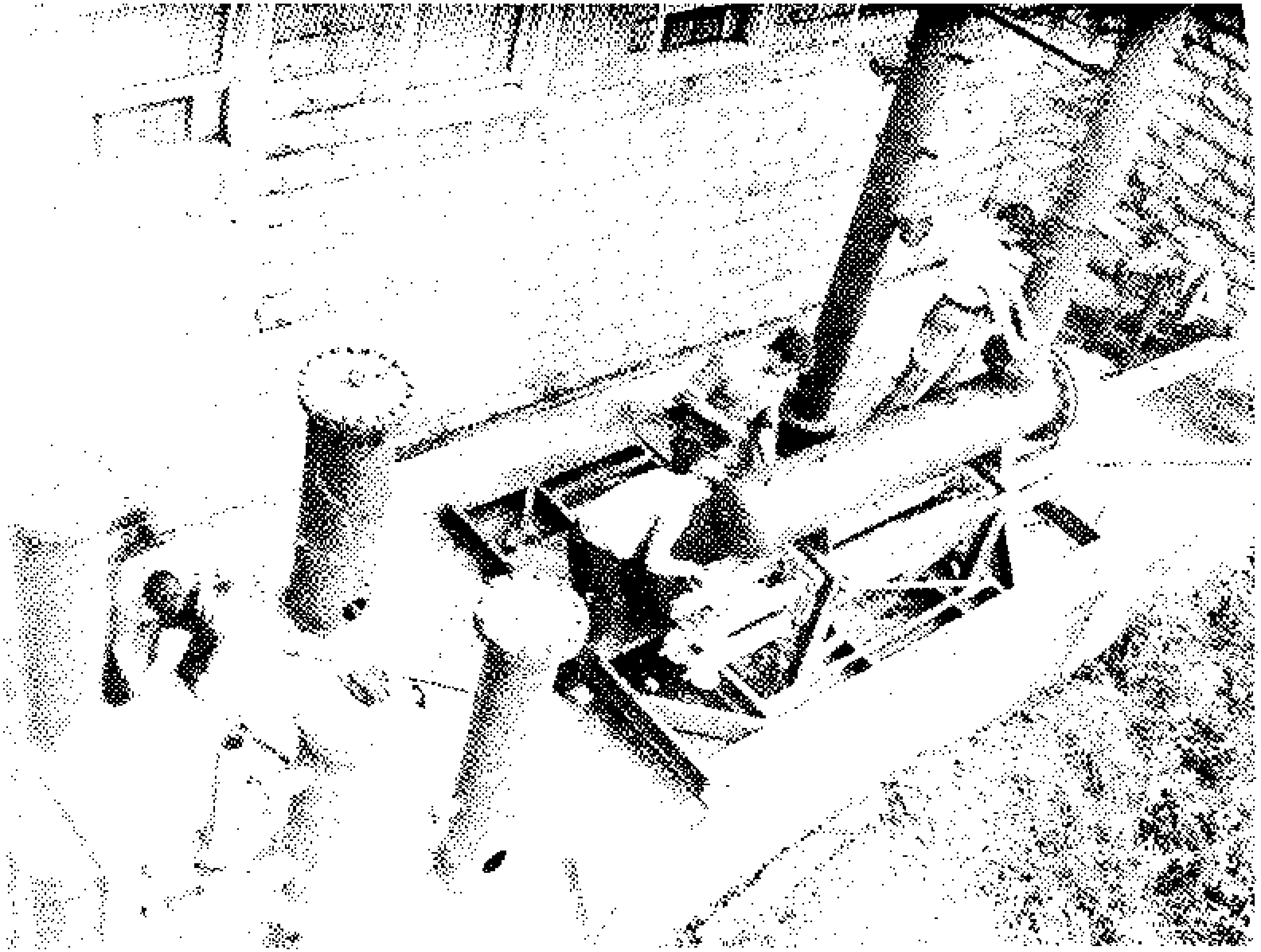

Figure 66. - Purification pilot plant under construction.

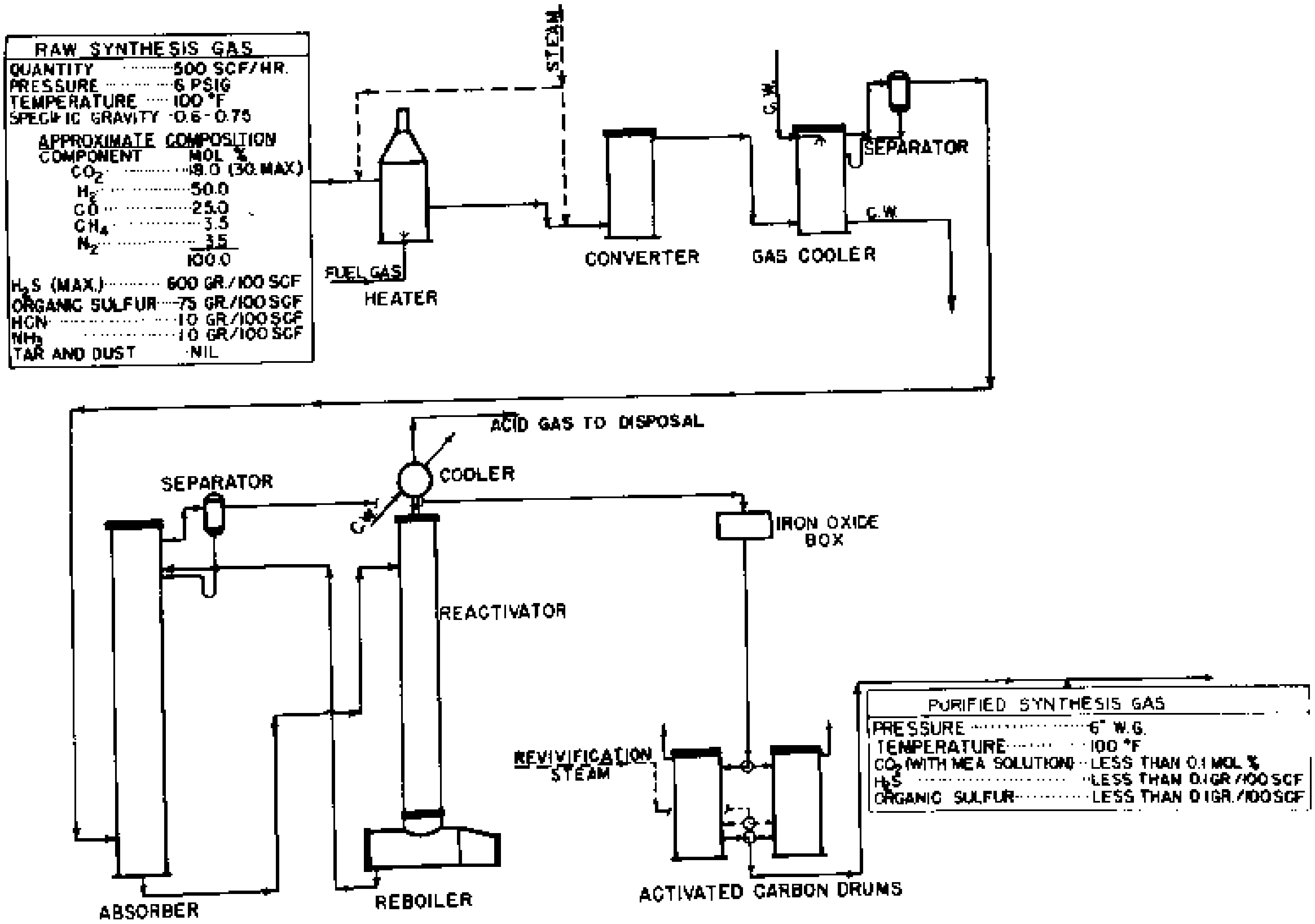

Figure 67. - Synthesis-gas purification pilot plant (500 S.C.F.H.).

It was assumed that 26 pounds of coal would be required per thousand cubic feet of synthesis gas and that 800 cubic feet of such gas would be required per gallon of product.

Operating costs for gas purification in this table include removal of hydrogen sulfide by a scrubbing process, final removal of residual hydrogen sulfide by iron oxide, and removal of organic sulfur. These costs include depreciation, maintenance, and interest, as well as labor, chemicals, and utilities.

Net costs of purification are indicated which show the lowered costs resulting when credit is taken for sulfur recovered from the hydrogen sulfide scrubbing process. Sulfur, at the mines in Texas or Louisiana, has been quoted at $18 per long ton f.o.b. railroad cars and $19.50 per long ton f.o.b. barges. Freight costs to most of the major coal-producing areas exceed $7.00 per long ton. If the hydrogen sulfide is burned to sulfur dioxide (as in sulfuric acid manufacture) and two thirds of the heat evolved is recovered to produce steam having a value of 30 cents per thousand pounds, a steam credit of about $2.50 per long ton of sulfur results. Combining the f.o.b. cost of sulfur at the point of production, a typical freight cost to a coal-producing area, and the steam credit, a value of $29 per long ton of sulfur could be assumed. How much of such value would have to be discounted in order to afford incentive for a sulfur-consuming plant to locate adjacent to the liquid fuels plant is open to question. For this reason, net costs of gas purification are shown, based on various values for sulfur (and steam) credits. It is interesting to note that if a value for sulfur and steam credit equivalent to $25 per long ton of sulfur could be assumed, it would be advantageous to use a high sulfur coal.

TABLE 16. - Estimated cost of synthesis-gas purification

Sulfur in coal ....percent	1	3	5
Investment in purification equipment	$3,865,000	$5,447,000	$6,688,000
Recoverable sulfur/day ....short tons	38	118	195
Total cost of sulfur removed per ton of coal gasified	$0.745	$1.15	$1.52
Net cost of purification per short ton of coal gasified, based on various sulfur credits[1]			
Credit per long ton sulfur recovered, percent	1	3	5
$ 5	$0.71	$1.03	$1.32
$10	.67	.91	1.12
$15	.62	.79	.92
$20	.58	.67	.71
$25	.55	.55	.51

[1] Sulfur credits include credit for heat recovered from sulfur conversion process, if any.

Purification Pilot Plant

A gas-purification pilot plant is under construction (see figs. 66 and 67). This plant will include an absorber column for removing the major proportion of the hydrogen sulfide, a stripping column for removing the hydrogen sulfide from the scrubbing liquor, a gas heater and catalyst vessel for catalytic removal or conversion of organic sulfur, and active carbon vessels for use in removing gum formers and organic sulfur which may not be removed catalytically. A moving-bed filter for removing dust from gas also is being constructed. Design was based on German shaft filters.

Operation of this pilot plant should yield information of value in preparing revised cost estimates and should determine the effectiveness of gas-purification methods developed in the laboratory.

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