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

	Paste oil	H.O.L.D.	L.O.B.	Middle oil	Naptha	Gasoline
Insoluble materials..wt. percent	8.					
Benzene-insol.	8.5	22.6	Nil	-	-	-
Pet. ether-insol.	12.3	29.2	do.	-	-	-
Density						
Sp. gr. ....60/60° F.	1.126	1.274	1.056			
°A.P.I.	-	-	-	10.8	16.4	48.5
Distillation................°F.						
I.B.P.			570	313	209	122
10 percent....................			658	532	350	178
30 percent....................			685	565	412	217
50 percent....................			708	582	443	245
70 percent....................			742	596	473	273
90 percent....................			-	614	513	313
E.P.			-	643	565	355
Recovery......................				98.2	983	97.6
Percent at 500° F.				5	84	-
Percent at 620° F.	12.9	13.0	4	93	-	-
Chemical analyses:						
Tar acids.....................				21.7	39.2	3.5
Tar bases.....................				5.0	4.6	1.5
Olefins.......................				19.8	12.7	20.8
Aromatics.....................				46.9	30.0	16.3

The high moisture content of this coal taxed the capacity of the grinding and drying system and from the beginning determined the rate of coal input to the hydrogenation unit. A maximum drying rate of only 3 to 4 tons of raw coal per hour was attained, requiring around-the-clock operation to furnish the minimum of dry coal for acceptable converter performance. Even at these low rates, the temperature of the gas at the inlet of the mill had to be 650° F., and the coal moisture could be reduced only to 8 to 10 percent.

The paste containing 28 to 32 percent m.a.f. coal was thinner than normal paste. However, minimum liquid-velocity requirements through the converters did not permit reduction of paste-oil quantities, and more coal for thickening of the paste could not be obtained from the coal-preparation system. The high moisture content of the prepared coal led to foaming in pastemaking operations. This tendency was overcome by reducing the temperature of the paste oil to 190° F.

Three percent (by weight of coal) of dry copperas ($FeSO_4 \cdot 7H_2O$) was added to the coal before grinding and drying. When converter performance became erratic and uncertain, the quantity of copperas was reduced by one-half, and ammonium molybdate was added at a rate of 0.2 percent Mo. The effects, if any, of the molybdenum were completely clouded by the extent to which irregular reactions had developed in the converters at the time of change in catalyst and by an inadvertent reduction in coal rate brought about by malfunctioning of the coal-delivery system.

Conversion to light oils, though low on the basis of the total organic coal substance, was on the order of 58 to 60 percent based on the carbon content of the coal. Liquefaction was consistent with the amount of relatively inert substance of the coal, as indicated by petrographic analysis. Gasification was not excessive, considering that over one-third of the gas produced was CO_2.

Performance of Equipment

In general, processing equipment performed well during this run. Steam tracing of instruments and winterizing of the plant, as indicated by the experience with the winter run on Lake DeSmet coal, proved adequate. No trouble was experienced with frozen instruments and controls. Improved operation was obtained from nearly all the injection pumps. Gasket leaks at valve assemblies were virtually eliminated by changeover from aluminum gaskets to copper. Stellite inserts were used successfully for the first time as valve seats. Packing replacements were low; only six rods were repacked to prevent leaks. The largest maintenance item on the injection pumps was replacement of ball-type valves. More than normal failure of valves seemed to occur during this run.

No difficulties were encountered in operation of the paste preheater. For the first time, no scaling or deposition of solids occurred in any of the tubes of this radiant-type heater. Better heat transfer was evinced by lower tube temperatures for the same heat load as for previous runs. Whether this good performance was due to characteristics of coal and paste oil, better firing, or the low solids content of the paste (38-42 percent) was not ascertained.

Inability of the coal-preparation unit to furnish enough coal steadily for proper operation of the converters caused considerable difficulty. Hot-spot reactions developed several times, necessitating quenching of excessive temperatures with cooling gas and pasting oil. The sudden coolings caused small leaks in flange joints which required operation at lowered pressures for sealing. To aggravate conditions, a short circuit developed in the thermocouples of the first converter, resulting in recording of the same temperature at all levels. Because of the unsatisfactory converter conditions, the run was terminated to make corrective changes. As the coal-preparation unit was unable to handle the requisite quantities of lignite for proper converter operation, it was decided to size the converters to the capacity of coal preparation. New liners, 17 inches in diameter, to replace the 21 1/2-inch liners of the converters, were ordered and received, but not in time for installation before the closing of the plant.

Removal of solids from the heavy-oil-let-down (H.O.L.D.) was more difficult than in previous operations. Apparently, either because of the character of the heavy oil and solids or because of the increase in the rate of feed, the efficiency of solids removal by centrifugation decreased appreciably. Low solids removal by centrifugation necessitated removal of the major portion of the solids made by flash distillation - an operation resulting in unusually high oil losses, which amounted to some 30,000 gallons more of heavy oil than made in the hydrogenation operations. The pitch made was very viscous and sticky, and three times during the operations the bottom head had to be removed for cleaning of the lower section of the flash drum. Improved flash nozzles performed well with little or no trouble from the coking experienced in previous runs.

Removal of Solids and Heavy-Oil Recovery

Multiple centrifugation

A limited number of experiments was done on the removal of solids by multiple centrifugation of streams. Two tests were performed wherein the filtrate from the Bird centrifuge, high in solids but free of large particles, was fed to the German DeLaval. In turn, the concentrate from the DeLaval was rerun in the Bird on the theory that agglomeration of solid had occurred in the DeLaval concentrate and that further solids could be removed in the Bird at a low oil loss. The first test was a batch operation in which the DeLaval concentrate was mixed with light-oil bottoms (L.O.B.) before recentrifuging. The second was continuous operation in which the DeLaval concentrate was fed to the Bird mixed with the fresh H.O.L.D. feed. The heavy oil used was a low solids H.O.L.D. from the Velva lignite run which had responded very poorly to centrifugation by the Bird. Results of the tests are given in table 12.

TABLE 12. - Summary of Bird-DeLaval series centrifugation operations, 24-hour basis

Centrifuge	Bird alone	Bird-DeLaval batch operation	Bird-DeLaval continuous
Feed.......................... gallons:			
H.O.L.D.	10,520	6,825	6,705
DeLaval concentrate..................	-	1,600	1,460
L.O.B.	-	3,250	-
Total............................	10,520	10,675	8,165
Solids in feed.....................lb.	17,070	17,725	13,940
Final concentrate:			
Solids.........................percent	52.2	47.8	55.0
Solids removed.....................lb.	1,790	3,775	3,930
Solids in H.O.L.D. feed.........percent	10.5	34.1	36.1
Oil loss: lb./lb. of solids	0.92	1.09	0.82
Final filtrate: solids..........percent	14.8	10.0[1/]	9.6

1/ Corrected for dilution effect of the L.O.B.

In these multiple centrifugation schemes the good feature of each centrifuge was utilized to best advantage, obtaining a low solids filtrate from the DeLaval and maximum oil recovery from the Bird. The capacity for solids removal by the combined operations was more than double that for the Bird alone. However, heavy oils vary with each coal and distinct hydrogenation operation and probably may not all respond to multiple centrifugation to the same degree. Additional experimentation with other oils is indicated.

Carbonization

Carbonization of solids bearing heavy-oil residues offers a means of increasing recovery of heavy oils in solids removal operations. The facilities at Louisiana, Mo., did not include carbonization or coking units. Consequently, to secure information on carbonization, some tests were made under contract with a commercial laboratory. Three tests were made on a modified Knowles sole-type coking oven. This was a batch-type oven but considered adequate for projection of yields to a continuous operation. The 3 tests were made on oils from Pittsburgh-seam coal and covered (1) H.O.L.D. (2) topped H.O.L.D., and (3) flash pitch. Analyses of the oven charge and product oils and yields obtained are given in table 13. Solids removal in all 3 cases was indicated to have been in the order of 99 percent of the amount of solids in the charge, and asphalt removal - 92.0 to 96.7 percent. Oil recovery exhibited a linear inverse relationship to the percent ash in the charge. Whether this relationship was significant or just fortuitous was not determined.

TABLE 13. - Analyses of charge materials and product oils; yields obtained in coking Pittsburgh-seam H.O.L.D. and flash pitch

	H.O.L.D.	Flash pitch	Vacuum-topped H.O.L.D.
Analyses:			
Carbon..................percent ash-free basis	88.6	87.9	89.4
Hydrogen..................................do.	7.0	5.7	5.6
C/H ratio......................................	12.7	15.4	16.0
Insol. in benzene.....................percent	20.6	44.6	47.5
Insol. in pet. ether......................do.	31.4	65.8	66.7
Ash...do.	13.1	27.9	18.8
Yields..................percent of total charge:			
Gasification...................................	4.4	4.6	3.2
Oil..	51.8	9.3 1/	26.1
Coke...	40.0	78.2	70.5
Loss...	3.8	7.9 1/	2.7
Oil recovery.......................wt. percent:			
(No-loss basis)................................	67.8	25.9	51.1
Solids removed.................................	99.2	98.8	99.5
Asphalt removed................................	90.5	93.2	95.8
Oil loss per lb. solids removed................	1.25	0.93	0.75
Oil analyses..........................percent:			
Insol. in benzene..............................	0.3	3.6	0.6
Insol. in pet. ether...........................	1.9	10.0	3.0

1/ Low oil yield and high loss owing to incomplete recovery of oils from a plugged line in recovery system.

A comparison of various methods and combinations of methods for solids removal, with and without carbonization, is presented for Pittsburgh-seam coal in table 14, based on typical hydrogenation operations on this coal at Louisiana, and the experimental carbonization data. The improvement effected by carbonization of residues is at once evident. Centrifugation with and without carbonization of the concentrate was by far the most efficient from the standpoint of oil economy. However, centrifugation as practiced in Louisiana was not in itself sufficient for balanced solids removal nor did it effect an asphalt purge. The distillation methods with or without carbonization of pitch, as expected, were effective in removing asphalts, the purge being some 130 percent of the asphalt made on typical operations with Pittsburgh-seam coal. Oil losses in all thermal cases were prohibitively high. A combination process involving centrifugation for low oil losses and carbonization for asphalt balance is indicated.

TABLE 14. - Comparison of methods for removal of solids; liquid-phase hydrogenation of Pittsburgh-seam coal

	H.O.L.D. analyses percent		Oil and asphalt loss	
	Solids	Asphalt	Lb./lb. solids removed	Percent on m.a.f. coal
Louisiana operations:				
Centrifugation of H.O.L.D. (Bird centrifuge)............	17.7	10.0	1.12	13.8
Flash distillation of H.O.L.D..	17.7	10.0	1.91	23.7
Experimental:				
Vacuum topping of H.O.L.D.	20.6	10.2	2.14	26.6
Carbonization of H.O.L.D.	20.6	10.8	1.25	15.5
Vacuum topping + carbonization of pitch....................	20.6	10.2	1.35	16.8
Estimated:				
Centrifugation (Bird) + carbonization of concentrate.......	17.7	10.0	0.36	4.5
Flash distillation + carbonization of pitch...............	17.7	10.0	1.46	18.1

	Asphalt purge (bal. solids oper.), percent on hydro make	Oil recovery, percent	Anal. recovered oil, percent	
			Solids	Asphalt
Louisiana operations:				
Centrifugation of H.O.L.D. (Bird centrifuge)............	Nil	95.2	13.2	11.8
Flash distillation of H.O.L.D..	138	61.0	1.7	2.1
Experimental:				
Vacuum topping of H.O.L.D.	126	35.6	.2	1.1
Carbonization of H.O.L.D.	137	67.8	.3	1.6
Vacuum topping + carbonization of pitch....................	122	65.4	.3	1.5
Estimated:				
Centrifugation (Bird) + carbonization of concentrate.......	Nil	98.7	12.8	11.5
Flash distillation + carbonization of pitch...............	137	70.2	1.7	2.2

Precoat Filtration

Just before the plant was closed initiatory experiments on filtration of H.O.L.D. as a means of removing solids were started in Louisiana. Several exploratory tests were made with a precoat rotary filter of 9 sq. ft. surface, acquired on a rental basis. Time did not permit necessary alterations for attaining optimum operating conditions, and the capacity reached was considerably lower than that indicated by laboratory filter-leaf tests. Data obtained are presented in table 15.

TABLE 15. - Data on experimental filtration of Velva lignite H.O.L.D.

	Precoat filter Avg. (2 tests)		Filter leaf		
Temperature of filtration .... °F.	250		280	345	365
Capacity .... gal./sq.ft./hr.	2.4		6	8.3	9.9
	Solids	Asphalt	Solids		
Analyses percent:					
Feed	19.8	6.0	15.8		
Filtrate	4.8	10.9	1.2		
Residue	59.5	7.4	48.6		
Benzene-sol. oil recovery .... percent		87.0	(81.0)		
Loss .... lb./lb. solids removed		0.64	(1.0)		
Asphalt purge		Nil	-		

Oil losses are low and probably can be reduced to equal centrifugation - carbonization losses, by carbonization or hot washing of the residue. However, as with centrifugation, an asphalt purge was not effected.

Operations - Vapor Phase

Vapor-Phase Run 5, Illinois No. 6 (Neutral oil) coal, Pittsburgh-seam coal and Velva lignite

Immediately after liquid-phase operations on Velva lignite ceased, the vapor-phase unit was prepared for processing the light oils accumulated from the lignite and last year's run on Pittsburgh-seam coal. Meanwhile, the Illinois No. 6 oils submitted to industry under cooperative agreement for extraction of tar acids and bases were returned and the vapor phase operation was initiated by processing of this oil. A total of 390,000 gallons of light oils from the 3 coals was processed in 45 days. Mechanical difficulties necessitating shutdown of the unit for repairs extended the run from the early part of January into April. Approximately 400,000 gallons of 83 octane (Research, clear) motor gasoline was produced.

Typical operating conditions and yields for each of three charge stocks are given in tables 16 and 17 and analyses of feed and product streams in table 18. Except for the Illinois No. 6 neutral oils, the feed stocks did not differ significantly from those previously processed, and the gasoline yield was essentially of the same magnitude and quality for both coals as obtained on previous operations. The Illinois stock was composed mainly of light-boiling materials low in tar acids and aromatics. High once-through yields at low temperatures were attained. A lower octane gasoline was produced than that from normal stocks. Although gasification was high the hydrogen consumption was low because of the hydrogen-rich character of the feed.

TABLE 16. - Typical operating conditions; vapor phase run 5

	Illinois #6 Neutral Oil	Pittsburgh Seam	Velva Lignite
Pressure..........................p.s.i.:			
Stall inlet.........................	9,900	10,000	10,000
Stall outlet........................	9,100	9,400	9,350
Converter temp. ....°F.:			
Average.............................	879	893	892
Maximum.............................	909	920	930
Feed injection:			
Gallons per hour....................	1,020	945	945
Lb./cu.ft./hr., total oil............	72.9	73.0	72.1
Lb./cu.ft./hr., > 375° F.	46.5	68.1	64.9
Recycle....................vol. percent	36.9	64.1	51.8
Gas flows.........cu.ft./lb. feed consumed			
Feed-injection gas..................	56.0	129.4	94.9
Cooling gas.........................	8.8	20.8	13.6
Total to stall......................	64.8	150.2	108.5
H_2.........................percent	86.7	83.6	-
Make-up H_2 gas..........................	7.4	13.8	10.6
Catalyst:			
Type................................	K-536	K-536	K-536
Volume.........................cu.ft.	100	100	100

TABLE 17. - Typical yields; vapor-phase run 5

	Illinois #6 Neutral Oil	Pittsburgh Seam	Velva Lignite
Yields......wt. percent on total oil feed			
Gasification........................			
Carbon monoxide, carbon dioxide......	-	0.2	-0.8
Net hydrocarbons....................	5.3	6.3	5.3
Gasoline............................	55.7	31.7	38.4
Recycle bottoms	38.5	64.1	55.1
Total oils..........................	94.2	95.8	93.5
Conversion of > 375° oils...wt. percent			
Gasoline............................	31.3	26.8	33.5
H.C. gases..........................	8.3	6.8	6.6
Total...............................	39.6	33.6	40.1
Yields on converted material wt. percent:			
Gasoline............................	79.0	79.6	83.5
H.C. gases..........................	21.0	20.4	16.5
Yields on feed consumed			
Gasoline..................wt. percent	90.6	88.3	85.5
H.C. gases..........................do.	8.7	17.5	11.8
Gasoline..................vol. percent	100.1	105.5	102.6
Space-time yields..........lb./cu.ft./hr.			
Total gasoline......................	40.6	23.1	28.6
Gasoline from > 375° F. feed...........	14.6	18.2	21.5
Reaction hydrogen....................			
Wt. percent on feed consumed..........	2.2	4.5	4.0

TABLE 18. - Typical analyses of feed and product streams; vapor-phase run 5

	Illinois No. 6 (neutral oils)				Pittsburgh seam				Velva lignite			
	Virgin feed	Feed blend	Recycle bottoms	Finished gasoline	Virgin feed	Feed blend	Recycle bottoms	Finished gasoline	Virgin feed	Feed blend	Recycle bottoms	Finished gasoline
Gravity....°A.P.I..	37.6	34.6	26.2	57.5	21.3	20.9	21.5	52.9	22.8	23.3	23.3	54.2
Distillation..°F.:												
I.B.P.	134	124	334	87	129	185	395	96	124	156	408	92
10..............	182	185	413	131	237	391	416	130	202	382	432	136
30..............	255	287	418	173	416	433	434	190	406	440	448	186
50..............	325	397	432	219	478	463	458	230	496	472	472	227
70..............	403	427	446	273	537	512	501	264	552	512	506	270
90..............	462	479	487	346	590	570	561	320	628	580	574	324
E.P.	564	541	559	387	618	602	608	374	666	630	642	392
Percent recovered					97.5	97.5	97.7	98.5	-	97.5	98.0	98.0
Percent at 375°F.	62.0	42.0	1.0	-	22.0	8.0	0	-	25.0	9.0	0	-
Chemical analyses..												
....vol. percent:												
Tar acids........	4.8	3.5	1.3	1.3	23.0	9.4	1.8	1.6	24.8	12.6	2.4	0.8
Tar bases........	1.7	1.2	0.4	1.6	5.2	2.2	.8	1.0	2.8	1.4	2.4	.8
Olefins..........	20.0	14.0	3.7	2.8	17.9	6.1	3.1	1.4	20.8	11.5	4.5	3.3
Aromatics........	24.4	36.9	58.2	22.1	31.7	48.2	56.3	27.8	29.9	49.1	61.8	27.2
Ultimate analyses..												
.........percent:												
Carbon...........	85.8	86.8	88.5	86.1	85.9	87.2	87.8	86.4	85.4	86.9	88.4	86.3
Hydrogen.........	12.4	12.0	11.3	13.7	10.5	11.2	11.3	13.4	10.6	10.9	10.8	13.4
Nitrogen.........	.12	.1	.1	.04	.9	.4	.1	.1	.4	.3	.1	.02
Sulfur...........	.15	.1	Nil	.04	.1	.06	Nil	.03	.4	.3	.3	.03
Oxygen (by diff.)	1.5	1.0	.1	.1	2.6	1.1	.8	.1	3.2	1.6	.4	.3
Octane No. (clear).												
Motor method.....				72.4								
Research.........								83.6				83.0

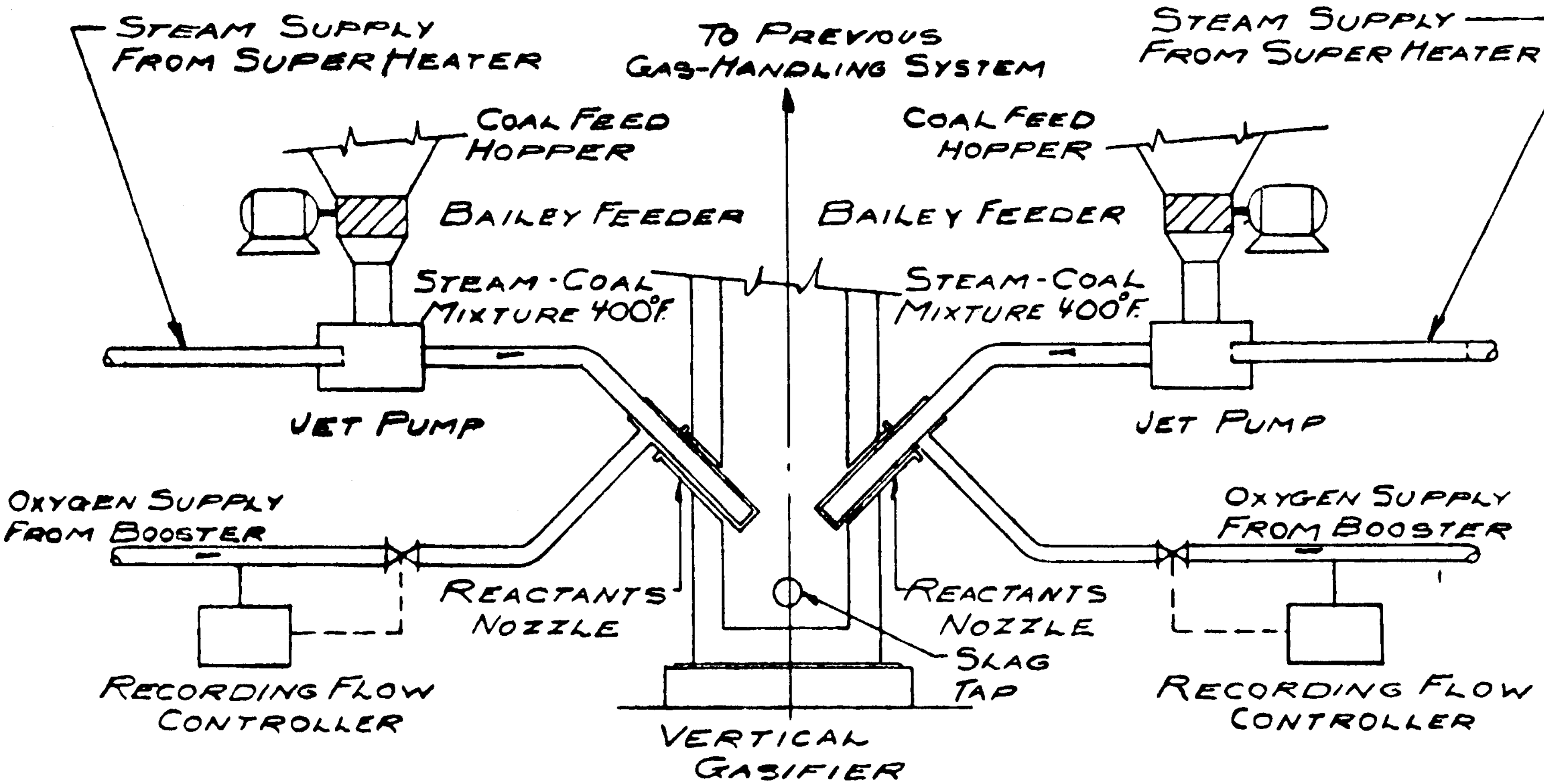

Figure 32. - Vertical gasifier, two-burner system.

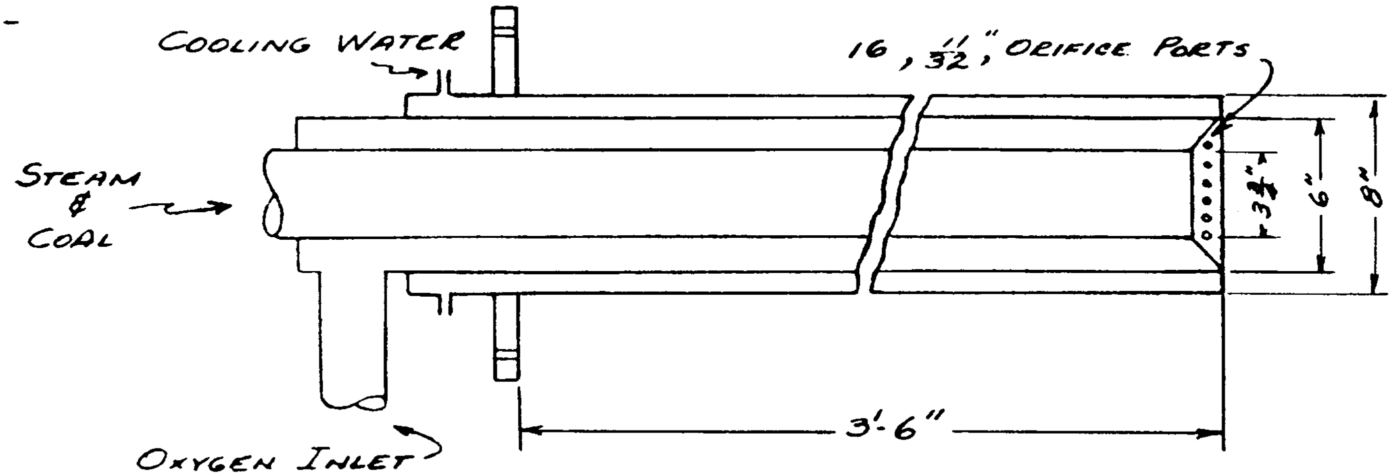

Figure 33. - Reactants nozzles, vertical gasifier.

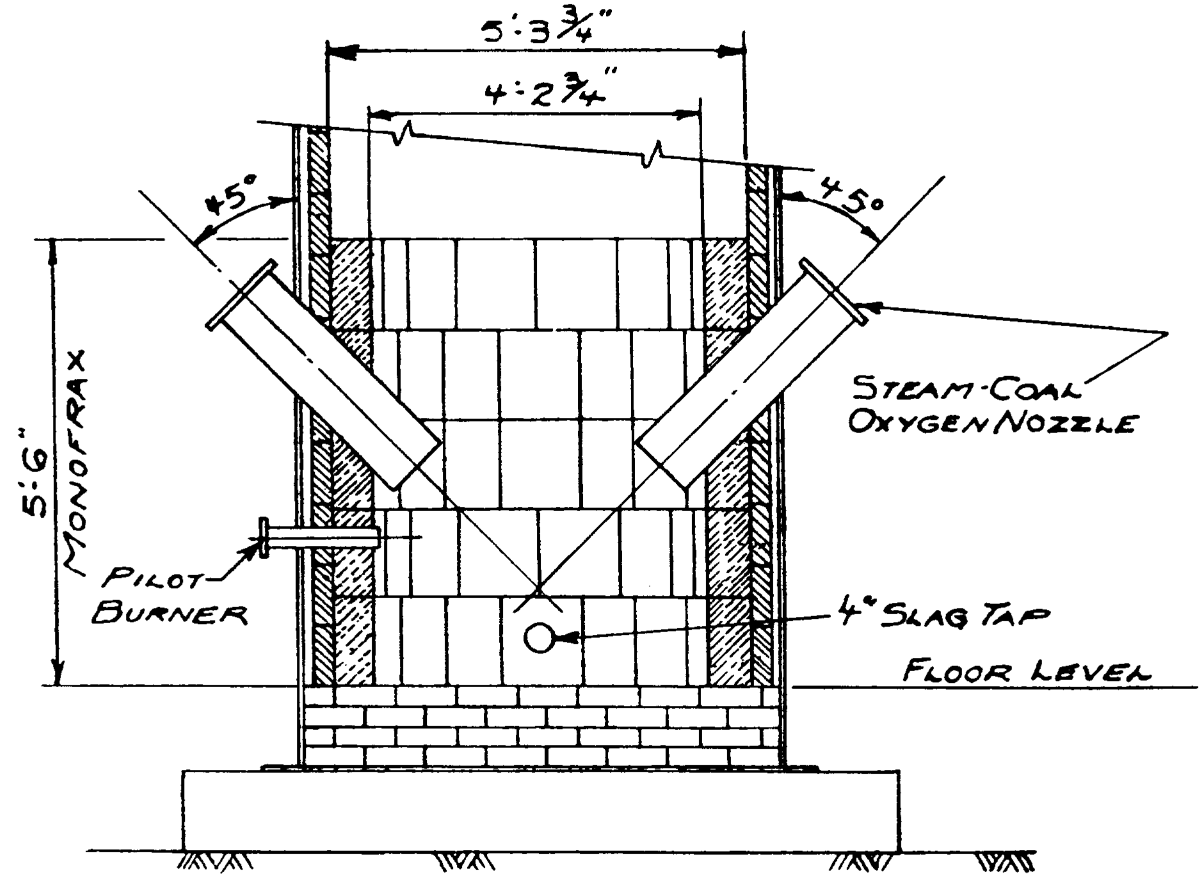

Figure 34. - Lower section, vertical gasifier two-burner system.

Gas-Synthesis Demonstration Plant

Coal Gasification

The installation of the second reactants nozzle as mentioned in the 1952 Annual Report was completed in May, and the vertical coal gasifier and purification equipment were operated from May 20 to May 29. This was the final operation before the plant was put into standby condition.

The vertical gasifier and its auxiliaries were revised extensively in preparation for run R-5. The most important change was addition of a second reactants nozzle similar in every respect to the original, both as to dimensions and axial orientation within the gasifier, but firing diametrically opposed to the original, single burner. As both burners were of the same size, the flow of oxygen, steam, and coal through each and the velocities of the reactants were reduced to half those obtaining in previous gasifier runs when only one burner was used.

The superheater coil was fed with steam at 150 p.s.i.g., the quantity being controlled by a recording flow controller. The superheater was gas-fired to maintain approximately 1,000° F. steam temperature. The superheated steam was divided by means of critical flow orifices into two branches, each leading to its own coal jet pump. From each leg of the coal bin powdered coal was fed by a constant-speed, star-wheel feeder into a weigh hopper. From the weigh-hopper coal was discharged into a feed hopper from which it was constantly withdrawn by a Bailey-star-wheel feeder equipped with variable-speed drive and recording tachometer. The Bailey feeder discharged the coal into the jet pump, where the superheated steam jet picked it up and discharged it into the gasifier through the reactants nozzle. The steam-coal mixture entered the gasifier at approximately 400° F.

The dual batch coal-weighing and continuous-feeding system was completely automatic, with a Syntron vibrator on each weigh hopper insuring accuracy of coal measurements. All the relays for this system, as well as for the other automatic controls, were moved from previous exposed locations into dust-tight enclosures. The oxygen supply from the Nash booster was fed through two recording flow controllers, each leading 1 of the 2 reactants nozzles. The coal-steam-oxygen feeding system is shown schematically in figure 32 while figure 33 shows the principal dimensions of the two reactants nozzles.

For a vertical distance of 6 feet from the floor or slag hearth, the gasifier was relined with 6-1/2 inches of Monofrax (fused alumina) shapes backed with 2-1/2 inches of high-temperature insulating brick. No chanes was made to the rammed alumina lining above this reaction zone. Figure 34 shows the new brickwork and the orientation of the reactants nozzles. Using the gas-fired standby burner, drying of the new brickwork was begun on May 13, after which the temperature of the reaction zone of the gasifier was gradually increased to 1,600° F. on May 20. During this period leaks which developed in the waste-heat boiler were repaired. The boiler was operated at 50 p.s.i. instead of 250 p.s.i., but when further leaks developed during the gasifier run, the steam was vented to the atmosphere.

The run was begun 5:50 p.m. on May 20, 1953, and continued substantially without interruption until the planned shutdown at 11 a.m., May 29, for a total of 209 hours of operation. The prescribed run conditions were: coal, 1,900-2,000 pounds per hour; oxygen, 19 M cu.ft. per hour; steam, 1,800 pounds per hour at a temperature of 1,000° F. leaving the superheater coil. These conditions were maintained throughout the run with small adjustments being made in coal and oxygen to prevent the reaction-zone temperature from exceeding 2,600° F. This temperature was measured by two Rayotube heads. During the first day of the run leaks of equalizing inert gas from behind the diaphragms of the bin level indicators of both coal hoppers caused erratic coal feeding and inaccurate weighing. After repairs were made, operation of the gasifier was quite steady. The newly installed controls operated well, and few changes to auxiliaries were needed to insure continuous operation.

The coal for the run was Illinois No. 6 seam from Peabody Coal Co.'s No. 17 mine. Proximate analysis (dry basis) of that used during most of the run averaged 46 percent volatile matter and 8.5 percent ash. The ash from earlier samples of this coal had a fusion temperature of approximately 2,320° F.

Slag was tapped at approximately 2-day intervals on May 23, 25, 27, and 29, for a total of 5,000 pounds, of which the largest tap was 1,700 pounds. Optical pyrometer readings on the flowing slag indicated temperatures of from 2,250° to 2,350° F. No exterior heating of the slag tap hole was required nor was there any interruption of the gas-making process while slag tapping was in progress.

During the run there was evidence that the make-gas orifice meter was not recording properly, and in the last 24 hours, 2 independent checks were made against the rate of rise of the synthesis-gas storage holder - one with the compressors operating and one with them idle. These 2 tests indicated the production of 7,000 to 10,000 cu. ft. per hour more gas than the make-gas flowmeter indicated. At the end of the run, it was found that the make-gas orifice may have been installed in reversed position, although this is not certain. A test showed that reversing the orifice would have made the meter read about 10 percent low. Accordingly, the results in table 19 are presented in three separate calculations: (1) For a period of about 2-1/2 days in which the operation was steady and smooth, with the make gas calculated from the orifice meter reading; (2) was calculated for the last 2 days of the run with the make-gas rate taken from the rate of the holder rise; (3) was calculated for a period of about 5 days, applying a flat 10-percent correction factor to the orifice meter. The only real difference between the 3 sets of figures is the make-gas rate and the figures that depend upon it. The gasifier conditions and the gas composition are substantially constant. If the make-gas meter is correct, the results in period A are valid and show a conversion of 87.6 percent of the inlet carbon at an oxygen-coal ratio of 10.4 cu. ft. per pound. This is the conversion which was achieved in run R-4 at an oxygen-coal ratio of 9.7 cu. ft. per pound. The results in period B, based on the holder rise, show almost 90 percent conversion at an oxygen-coal ratio of 10.0, which is not too far divergent from the results of run R-4. Period C, covering 5 days operation with the flat 10-percent correction to allow for the reversal of the synthesis-gas orifice, shows the best results with a carbon conversion of over 93 percent at an oxygen-coal ratio of 10.4 cu. ft. per pound. In this case there were required 34.5 pounds of coal and 358 std. cu. ft. of oxygen per M std. cu. ft. of $CO + H_2$. There is no basis for making a choice among these 3 sets of calculations. It seems reasonably certain that the conversion is not less than 87.6 nor above 93.3. It is of interest to note that calculations based on kinetic equations derived by Batchelder and Busche indicate for these operating conditions and time of contact, conversions almost exactly equal to those calculated for periods B and C.

TABLE 19. - Summary of gasifier operations

Run No. R-5 Run start - 5:50 p.m., 5/20 Run end - 11 a.m. 5/29/53

Periods for data	A	B	C
From	6 p.m. 5/22/53	7 a.m. 5/28/53	6 p.m. 5/21/53
To	7 a.m. 5/25/53	9 a.m. 5/29/53	7 a.m. 5/26/53
Duration, hours	61	26	109
Raw coal rate....lb./hr. (5.5% H_2O)	1,936	1/2,010	1,937
Process oxygen rate........std. cu.ft./hr.(98.8% O_2)	19,230	19,300	19,000
Process steam rate..........lb./hr.	1,800	1,800	1,800
Temp. of coal-steam into gasifier.. °F.	380	387	385
Oxygen (100%) to dry coal..........std.cu.ft./lb.	10.4	10.0	10.4
Steam-dry coal ratio........lb./lb.	0.985	0.950	0.980
Synthesis gas........std.cu.ft./hr.	62,200	67,700	67,950
H_2 + CO..........do.	48,600	53,000	53,010
Syn.-gas analysis..........percent:			
CO_2	17.0	16.8	16.8
H_2	39.6	39.5	39.6
CO	38.5	38.8	38.4
Ratio of H_2/CO	1.03	1.02	1.03
Charged carbon gasified.....percent	87.6	89.8	93.3
Gasifier temperatures 2/.........°F.			
Exit gas	2,075	2,050	2,100
By water-gas "shift" equilibrium.	2,420	2,260	2,210
Ratios per M std.cu.ft. of CO + H_2			
(A) Dry coal.........lb.	37.7	36.1	34.5
(B) Oxygen (100%).....std.cu.ft.	391	360	358
Total steam, lb./hr. 3/	1,926	1,920	1,924
Steam consumbed, lb.	311	399	439
"Economic factor," (A + B/10)......	76.8	72.1	70.3

Special conditions, equipment changes, coal source and grind:

Period A: Synthesis-gas flow based on orifice readings that were later found to be low.
Period B: Synthesis-gas flow based on gas-holder tests.
Period C: Synthesis-gas flow based on orifice recalibration tests.

1/ Contained 5.1% H_2O.
2/ Gasification zone and exit gas temperatures are best estimate based on temperature instrument and thermal calculation data.
3/ Process steam plus coal moisture plus moisture in the oxygen.

TABLE 19. - Summary of gasifier operations (con.)

Run No. R-5 Period	A	B	C
Ratios per lb. dry coal			
Oxygen (100%)..........std.cu.ft.	10.4	10.0	10.4
Process steam introduced......lb.	0.985	0.95	0.98
Steam consumed...............do.	0.017	0.017	0.024
Synthesis gas..........std.cu.ft.	34.0	35.4	37.1
H_2 + CO....................cu.ft.	26.6	27.7	29.0
Ratios per std. cu.ft. oxygen (100%)			
Synthesis gas..........std.cu.ft.	3.27	3.55	3.58
H_2 + CO.......................do.	2.56	2.78	2.79
Gasifier temperatures...........°F.			
Base, RC-Pot 170 (E)............	Out	Out	Out
RC-Pot 170-1 (W)...........	do.	do.	do.
Wall, ascending,			
R-Pot 170-1................	2,225	2,180	2,200
R-Pot 170-2................	2,160	2,075	2,100
R-Pot 170-3................	2,000	1,965	1,980
R-Pot 170-4................	1,850	1,745	1,770
Exit gas, RTC 173..................	2,075	2,040	2,100
Gas to W.H. boiler, R-Pot 138......	1,600	1,590	1,600
No. Synthesis-gas analyses, from the mass spectrometer.....percent	15	5	24
CO_2............................	17.0	16.8	16.8
H_2.............................	39.6	39.5	39.6
CO............................	38.5	38.8	38.4
CH_4............................	.1	.1	.1
C_2H_6............................	-	-	-
N_2.............................	3.6	3.6	3.9
A.............................	.3	.3	.3
O_2.............................	.0	.1	.1
H_2S............................	.9	.8	.8
H_2 + CO........................	78.1	78.3	78.0
H_2S (Tutweiler).grains/100 cu.f.t	560.0	595.0	580.0
Gross heating value................ ....B.t.u./std.cu.ft.	259	259	258
No. Orsat syn.-gas analyses........ ....percent:	7	4	15
CO_2............................	19.1	18.6	18.4
CO............................	37.5	38.8	38.7
O_2............................	.0	.0	.0
Raw coal feed			
Avg. r.p.m. 122 and r.p.m. 123...	33	34	33
No. dumps......................	487	221	882
Avg. dump wt.	238	236	239
Avg. raw-coal rate...............	1,936	2,010	1,937

TABLE 19. - Summary of gasifier operations (con.)

Run No. R-5 / Period	A	B	C
Oxygen flow			
Orifice meters.........cu.ft./hr.	19,500	19,600	19,500
Temp. at orifice............. °F.	78	80	79
Press. at orifice........p.s.i.g.	9.0	9.0	9.0
Process O_2 flow................. ....std.cu.ft./hr. dry	19,250	19,300	19,200
100% O_2 flow...std.cu.ft./hr. dry	19,000	19,070	19,000
Process steam			
Steam temp. after superheater.°F.	1,010	1,010	1,020
Synthesis-gas flow			
Orifice meter..........cu.ft./hr.	63,870		69,740
Temp. at orifice.............°F.	81		80
Press. at orifice........p.s.i.g.	19.6		19.0
Sp. gr. ref. to air.............	0.704		0.702
Flow of dry syn. gas............. ....std.cu.ft./hr.	62,200	67,700	67,950
Recycled gas			
If-144................cu.ft./hr.	35,000	35,000	35,000
Carbon conversion			
Total carbon in coal......lb./hr.	1,252	1,330	1,277
Carbon in syn. gas............do.	1,098	1,193	1,191
Unconsumed carbon............do.	154	137	86
Percent of total carbon utilized.	87.6	89.8	93.3
Reacted steam..............lb./hr.			
From hydrogen figures:			
H_2 in dry syn. gas............	136.5	145.0	149
H_2 in dry coal................	82.4	91.6	88
H_2 from steam (by diff.).......	54.1	53.4	61
Equivalent reacted steam.......	487	480	549
From oxygen figures:			
O_2 in dry syn. gas............	1,905	2,080	2,071
O_2 in dry coal................	181	187	179
O_2 from process O_2	1,603	1,610	1,602
O_2 from steam (by diff.).......	121	283	290
Equivalent reacted steam.......	136	318	328
Total steam...............lb./hr.:	1,926	1,920	1,924
Ave. reacted steam...............	311	399	439
Unreacted steam..................	1,615	1,521	1,485
Thermal calculations:			
Thermal balance temp. ....°F.	2,075	2,050	2,100
Gasifier heat loss.............. ....MM B.t.u./hr.(by diff.)	0.483	1.033	0.706
B.t.u. efficiency (heating value of syn. gas/coal heating value)	68.3	69.0	72.0
Heat loss as a percent of the total heat in	2.2	4.3	3.1

TABLE 19. - Summary of gasifier operations (con.)

Run No. R-5 Period	A	B	C
Pressures:			
In gasifier...............in H_2O	-0.1	-0.5	-0.3
Out of washer cooler.........do.	-3.7	-3.2	-3.5
In pulv. coal bins...........do.	3.5	4.0	4.0
Into superheater coil...p.s.i.g.	31.5	31.5	31.5
Coal feed trans. piece, north... ....in H_2O	-30	-30	-30
Coal feed trans. piece, south... ....in H_2O	-20	-20	-20
Coal-steam line, north.......do.			
Coal-steam line, south.......do.			
O_2 at N. burner.........p.s.i.g.	2	2	2
O_2 at S. burner.............do.	2	2	2
Coal analysis			
(A) Analyses of composite coal samples taken during run.			
Sieve test, percent passing -			
60-mesh...............	100.0	99.8	99.9
100-mesh...............	99.8	99.1	99.6
200-mesh...............	97.9	94.1	97.1
325-mesh...............	89.5	88.2	92.8
Moisture..........percent	5.5	5.1	5.5
(B) Analyses of composite of coal used for run			
Moisture as received..... ....percent	8.3	6.8	6.8
Ultimate, dry percent:			
H_2....................	4.5	4.8	4.8
C......................	68.4	69.8	69.8
N_2....................	0.6	0.8	0.8
O_2....................	9.9	9.8	9.8
S......................	4.8	4.9	4.9
Ash....................	11.8	10.0	10.0
Net heating value........ ....B.t.u./lb.	11,894	12,270	12,270

Miscellaneous analyses

Feed gas to Girbotol 9 a.m. 5/28/53
Organic sulfur 19.8 gr./100 cu.ft.

Slag tap record

Date..................	5/23/53	5/25/53	5/27/53	5/29/53
Time..................	5 p.m.	2:30 p.m.	2 p.m.	8:30 a.m.
Amount.............lb.	810	1,720	1,420	1,120
Temperatures.......... (Optical)	2,350		2,250	

Examination of the gasifier after the run showed that the refractory had only minor imperfections. There were two areas at right angles to the plane of the burners and just above the slag level which had been definitely eroded. These areas were about 12 inches wide and 18 inches high and at the worst point were about 2 inches deep. It is postulated that the two flames intersecting would "butterfly" downward and toward the walls. There were two small areas of erosion directly under each burner and just above the slag line. These were much less severe and much less extensive than the two on the side walls. In addition, there was a shallow groove completely around the gasifier just above the slag level. It is believed that with a modification to reduce the sideward component of the flame, a satisfactory operation could be achieved over an appreciable length of time.

Gas-Purification Plant

The Raschig rings in the Girbotol absorber were completely removed, cleaned, and replaced before the plant was used for purifying gas produced from coal. After the synthesis-gas holder was filled with gas from the vertical gasifier on May 22, the gas compressor and Girbotol units were put on the line. The two iron oxide towers and two activated-carbon towers were put into service the following day. The carbon towers were reactivitated in succession by passing 600° F. superheated steam through them for 10 hours and thereafter one was reactivated every second day, thus giving each 4 days continuous service before reactivation. The purification run was concluded on May 29.

The daily operating summaries in table 20 show that there was little variation in operating conditions from day to day, and the quality of the gas produced was at all times satisfactory.

Gas-Purification Data

Carbon dioxide removal is accomplished in the Girbotol unit. The volume of carbon dioxide removal averaged 9,200 std. cu.ft. per hr. The hydrogen sulfide content was reduced in the Girbotol unit from 600 to 2.5 grains per 100 cu. ft. of gas. The inlet organic sulfur was 20 grains per 100 cu. ft. and at the outlet, 1-1/2 grains.

1. Girbotol Unit

The carbon dioxide and sulfur removal, calculated from amine flow and analysis, agreed fairly closely with the quantities calculated from gas flows and analyses, although not as closely as in previous gasification runs. It was not possible, however, to obtain information to explain this in the short time the unit was on stream.

The following table shows, to some extent, the effect of hydrogen sulfide content of the feed gas on the acid gas content of the rich amine solution:

H_2S per 100 cu.ft. per hr. in feed gas, grains	Rich amine (from absorber)		Lean amine (from reactivator)	
600 (for this run)	196 grains H_2S per gal. of solution	33 ml. CO_2 per ml. of solution	47 grains H_2S per gal. of solution	3.0 ml. CO_2 per ml. of solution
75 (for run previously reported)	52.3 grains H_2S per gal. of solution	45.9 ml. CO_2 per ml. of solution	29.6 grains H_2S per gal. of solution	3.7 ml. CO_2 per ml. of solution

TABLE 20. - Daily operating summaries; gas-purification plant

Date	5/23/53	5/24/53	5/25/53	5/26/53	5/27/53	5/28/53
Gas feed ... std.cu.ft./hr.	51,900	52,300	51,300	52,600	51,300	52,500
Purified gas ... do.	43,000	42,700	43,000	43,000	42,140	43,000
Feed-gas analysis:						
H_2 ... percent	40.3		39.9			
CO ... do.	37.0		39.0			
CO_2 ... do.	17.6	18.5	16.3	18.2	17.9	18.2
Misc. ... do.	5.1		4.8			
H_2S ... grains/100 cu.ft.	610	557	519	627	625	640
Ratio H_2:CO	1.08		1.02			
Purified-gas analysis:						
H_2 ... percent	48.6		47.6			
CO ... do.	44.7		46.5			
CO_2 ... do.	.30	.30	.10	.00	.1	.05
Misc. ... do.	6.4		5.8			
Total S ... grains/100 cu.ft.	0.010	0.000	0.000	0.000	0.004	0.000
Gas pressure to purification ... p.s.i.	370	370	370	370	370	370
Gas pressure leaving purification. do.	340	340	340	340	340	340
Girbotol:						
Amine (DEA) flow ... g.p.m.	44	44	43	43	43.3	43.3
DEA concentration in amine solution ... percent	37.6	37.7	40.3	37.1	40.1	38.7
Temp. gas to absorber ... °F.	83	86	89	87	86	81
Temp. gas from absorber ... do.	105	105	103	105	104	105
Temp. amine solution to absorber ... °F.	120	113	111	112	118	117
Temp. amine solution from absorber. ... °F.	165	163	161	159	164	165
Reactivator pressure ... p.s.i.	13.4	13.5	13.0	13.0	12.8	13
Reboiler steam pressure ... do.	32	32	32	31.6	32	32
Temp. gas from reactivator column ... °F.	172	169	181	174	187	201
Temp. gas from acid gas cooler.. do.	150	150	152	150	153	151
Temp. amine solution to reactivator ... °F.	187	185	193	186	202	222
Temp. amine solution from reactivator ... °F.	246	246	250	247	250	250
Steam usage on reboiler ... lb./hr.	1,720	1,720	1,720	1,710	1,720	1,720
Water to amine cooler ... g.p.m.	96	101	79	90	67	71
H_2S in gas from absorber ... grains/100 cu.ft.	2.5	2.1	4.2	1.7	1.4	2.2
Acid content of DEA:						
Rich (from absorber):						
H_2S ... grains/gal.	209	200	198	205.1	187.4	179.4
CO_2 ... ml./ml. sample	39	41.3	31.7	32.7	28.0	25.5
Lean (to absorber):						
H_2S ... grains/gal.	61	61	47.7	43.1	37.8	28.8
CO_2 ... ml./ml. sample	4.5	5.3	3.0	3.1	1.1	.8
Total raw gas to purification.. M c.f.	1,246	1,256	1,115	1,262	1,231	1,260
Total purified gas ... do.	1,032	1,025	935	1,032	1,136	1,032

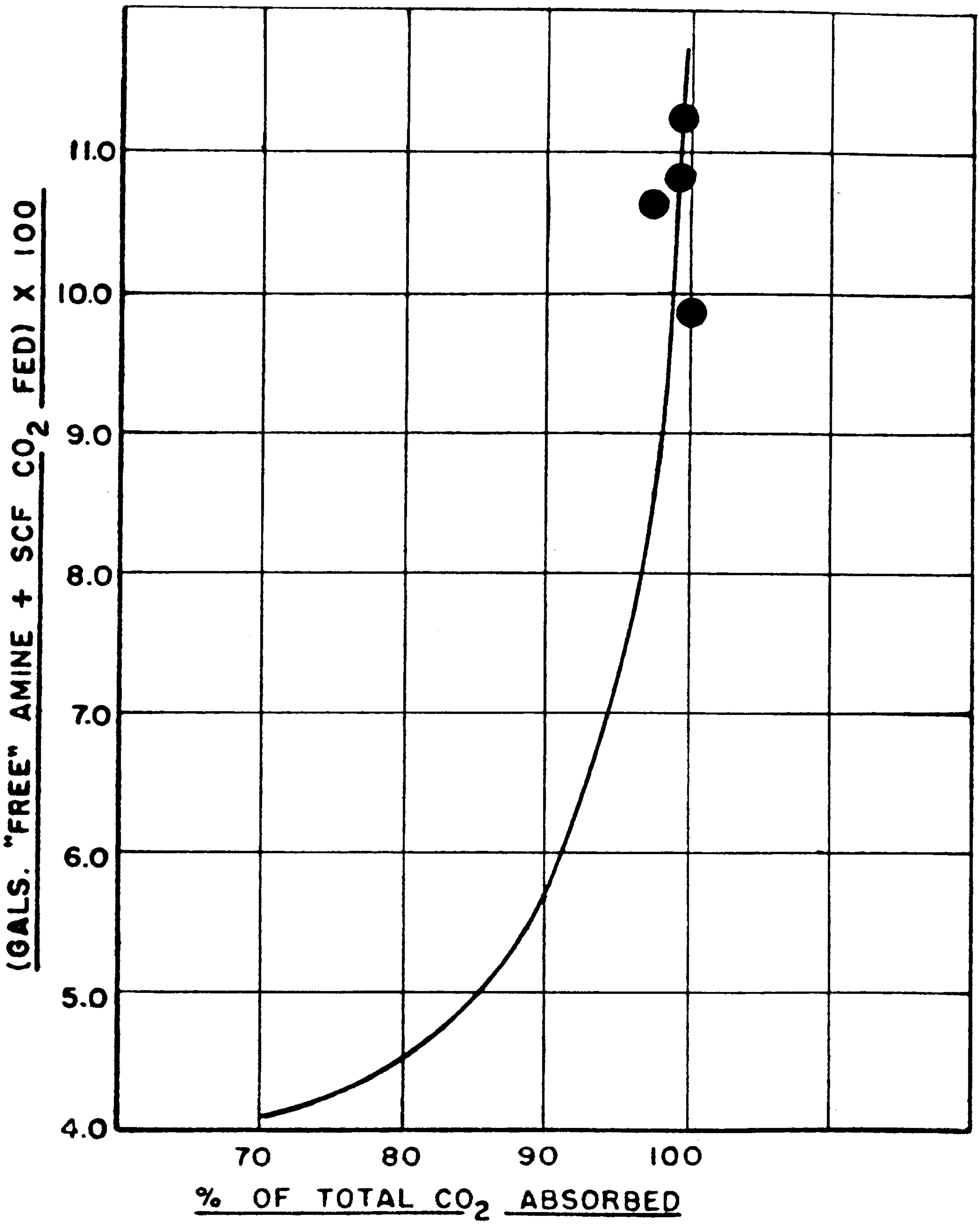

Figure 35. - Diethanolamine scrubber performance.

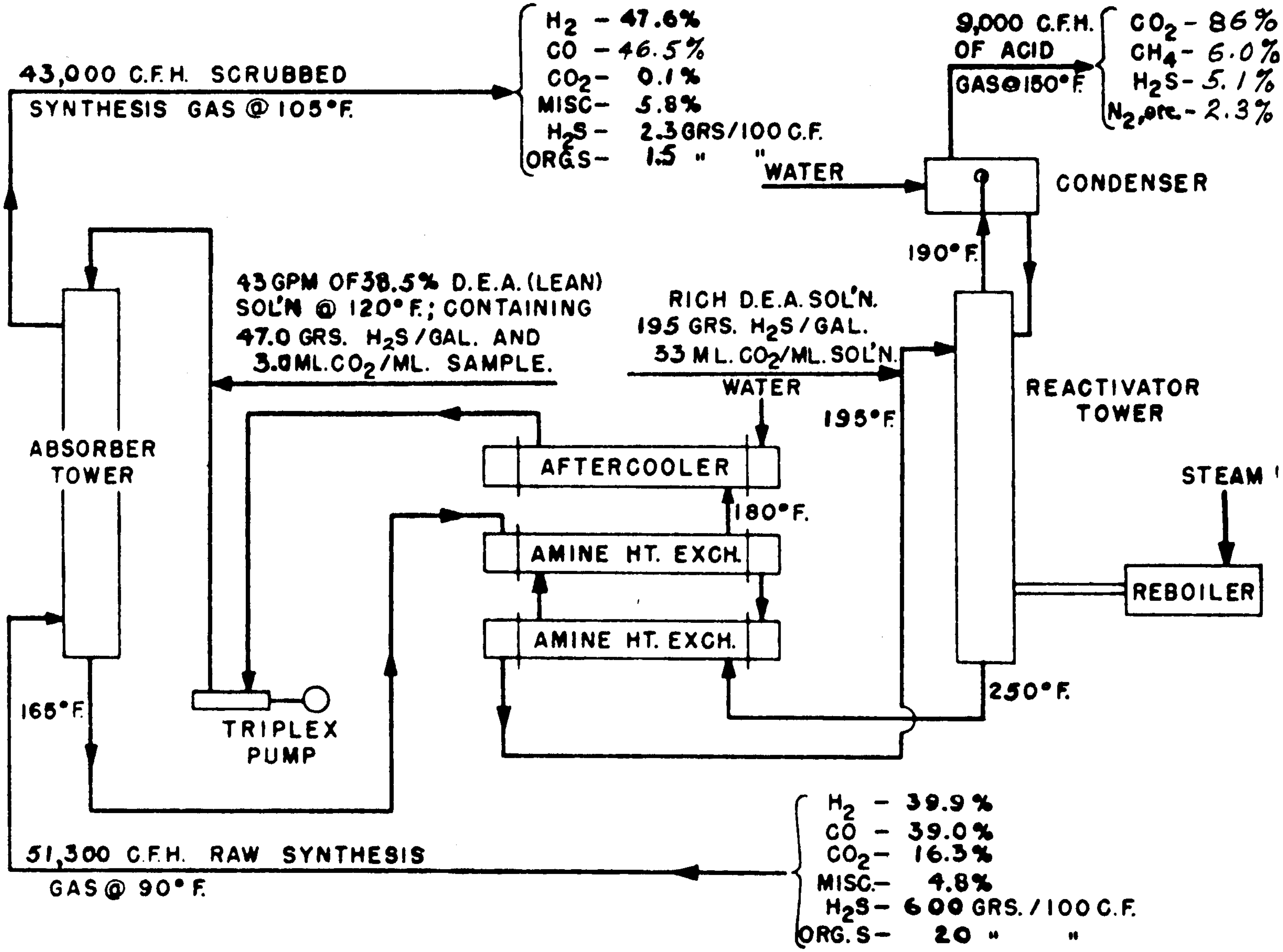

Figure 36. - Diethanolamine scrubbing - typical operation.