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PERFORMANCE OF A GAS-SYNTHESIS DEMONSTRATION PLANT FOR PRODUCING LIQUID FUELS FROM COAL

By R. G. Dressler and L. L. Hirst

UNITED STATES DEPARTMENT OF THE INTERIOR
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PERFORMANCE OF A GAS-SYNTHESIS DEMONSTRATION PLANT FOR PRODUCING LIQUID FUELS FROM COAL[1]

by

R. G. Dressler[2] and L. L. Hirst[3]

SUMMARY

The Federal Bureau of Mines designed, constructed, and operated a demonstration plant at Louisiana, Mo., to produce gasoline, liquid fuels, and organic compounds from coal by a modified Fischer-Tropsch gas-synthesis process. Satisfactory equipment was developed to produce and purify synthesis gas and convert it into the desired products. Coal or coke was gasified with oxygen and steam to make a gas consisting essentially of carbon monoxide, hydrogen, and carbon dioxide. Dust in the raw gas was removed by cyclone separation, water scrubbing, and electrostatic precipitation. Carbon dioxide, hydrogen sulfide, and organic sulfur were removed with diethanolamine, iron oxide, and activated carbon. The purified gas was passed over iron catalyst and was converted into synthetic liquids, which were then separated and fractionated into gasoline, diesel oil, other liquid fuels, and oxygenated organic compounds.

A 39-day continuous run demonstrated the feasibility of the process. Addition of 3 milliliters of tetraethylead per gallon of upgraded (or finished) gasoline gave a product with an octane rating up to 90 by the research method. After a caustic wash, the diesel oil was of a superior quality; its cetane number was 60 or above.

Approximately 1,500,000 standard cubic feet of synthesis gas was processed into about 25 barrels of gasoline and 150 gallons of diesel oil per day. Table 1 shows typical yield and distribution of synthesis products per 100,000 standard cubic feet of $CO + H_2$. Conversions of carbon monoxide and hydrogen to liquid products ranged from 80 to 90 percent.

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[1]Work on manuscript completed October 1960.

[2]Former chief of Gas-Synthesis Demonstration Plant, Fuels Technology Division, Bureau of Mines, Louisiana, Mo.; now chemical process consultant, San Antonio, Tex.

[3]Research director, Coal Research Center, Bureau of Mines, Morgantown, W. Va.

TABLE 1. - Yield and distribution of products from the conversion of 100,000 standard cubic feet of $CO + H_2$ at the Gas-Synthesis Demonstration Plant

Products[1]	Gasoline, gallons	C_3+ products, gallons	Gasoline, volume-percent	C_3 products, volume percent	Liquid products, volume-percent	$CO + H_2$ converted per barrel, thousand standard cubic feet	Product per cubic meter $CO + H_2$ converted	
							Gasoline, grams	C_3+ products, grams
Propane.....	-	30	-	20	-	140	-	21
Butane.....	13	-	13	-	-	326	10	-
Polymer gasoline..	29	-	30	-	-	145	28	-
C_5+ 400 end point.	55	-	57	-	-	76	51	-
Total gasoline.	97	97	100	63	79	43	89	89
Diesel oil.....	-	12		8	10	350	-	13
Coolant oil[2].....	-	13		9	11	326	-	15
C_3+ products...	-	152		100	100	27.5	-	138

[1]20 M std.c.f. of CO_2, 2.9 M std.c.f. of CH_4, C_2H_6, and 43 gal. of oxygenated organic compounds also produced.
[2]Include waxes.

Substantial quantities of catalyst disintegrated during the gas-synthesis runs. Although the catalyst retained its activity and synthesis reactions continued, the disintegration of catalyst was the major unsolved problem at the demonstration plant. Most of the mechanical difficulties were overcome, however, and a commercial unit probably could be designed, using information obtained at the demonstration plant and at the Bureau of Mines pilot-scale unit at Bruceton, Pa.

INTRODUCTION

The Federal Bureau of Mines constructed a demonstration plant at Louisiana, Mo., to produce gasoline and diesel oil from coal by a modified Fischer-Tropsch gas-synthesis process. This modified process yields a gas that is purified and synthesized into liquid hydrocarbons and oxygenated organic compounds. Several publications discuss the design and process development of the entire plant and the performance of the gasification and purification units from the beginning of operations in 1949.[4-6]

[4]Dressler, R. G., and Bircher, J. R., The Bureau of Mines Gas-Synthesis Demonstration Plant at Louisiana, Mo.: Pres. at ASME Petroleum Mechanical Engineering Conference, New Orleans, La., Paper 50-PET-9, Sept. 25-28, 1950.
[5]Kastens, M. L., Hirst, L. L., and Dressler, R. G., An American Fischer-Tropsch Plant: Ind. Eng. Chem., vol. 44, No. 3, March 1952, pp. 450-466.
[6]Markovits, J. A., and Hirst, L. L., Two American Coal-to-Oil Demonstration Plants: The Mines Magazine, November 1952, pp. 129-144.

Basic research and pilot-scale work were conducted by the Bureau of Mines at Bruceton, Pa.[7]

Engineers from abroad and technical representatives of petroleum and chemical companies visited the plant at Louisiana to keep informed on developments in coal gasification and in the production of synthetic chemicals and liquid fuels. The details of the design and development of equipment and the performance of the gasification and gas-purification phases of the process are given in the publications cited, but the results of integrating these phases with synthesis and refining have not been reported. This report briefly describes the plant and process and gives the performance of the integrated plant from initial operations in September 1951 until the program was terminated in 1953.

The experiments at Louisiana were terminated before a coal gasification system was developed that was reliable enough to permit extended gas-synthesis runs with the integrated plant. At the beginning of some of the synthesis runs limited quantities of gas made directly from coal were available in the gas-storage system, but gas for the trials of the integrated plant was made from coke instead of from coal. The purified gas from either source was virtually the same, however, and was satisfactory for use in developing the equipment and establishing the feasibility of the overall process. Gas for most of the trials of the purification system also was produced from coke. Before the work at Louisiana was terminated, however, the purification system has successfully purified gas produced directly from coal.

DESCRIPTION OF GAS-SYNTHESIS DEMONSTRATION PLANT AND PROCESS

The Gas-Synthesis Demonstration Plant, commonly called the Fischer-Tropsch Demonstration Plant, comprised the following process units: (1) Oxygen plant, (2) coal and coke gasification, (3) synthesis-gas compression and purification, (4) gas-synthesis section, and (5) synthetic liquid products refining and upgrading. Maximum design output was 60 barrels per day of finished product, including approximately 50 barrels of gasoline. Figure 1 shows all the main operating units except a catalyst plant that was auxiliary to the process. A brief description of the process follows. Additional details are presented later in this report.

Figure 2 is a flowsheet of the process as originally installed and operated for the first three of the four runs described later in this report. Coal is crushed in a ring-type crusher, pulverized in a spring-loaded ball-race mill, collected in a cyclone separator and bag filters, and stored. The powdered coal together with oxygen and superheated steam is fed to a gasifier. Raw synthesis gas from the gasifier is passed successively through the waste heat boiler, a cyclone separator that remove most of the entrained dust and fly ash, and the washer-cooler that removes almost all of the remaining solids and reduces the gas temperature to 100° to 150° F. Containing less

[7] Crowell, J. H., Benson, H. E., Field, J. H., and Storch, H. H., Fischer-Tropsch Oil Circulation Processes: Ind. Eng. Chem., vol. 42, No. 11, November 1950, pp. 2376-2384.

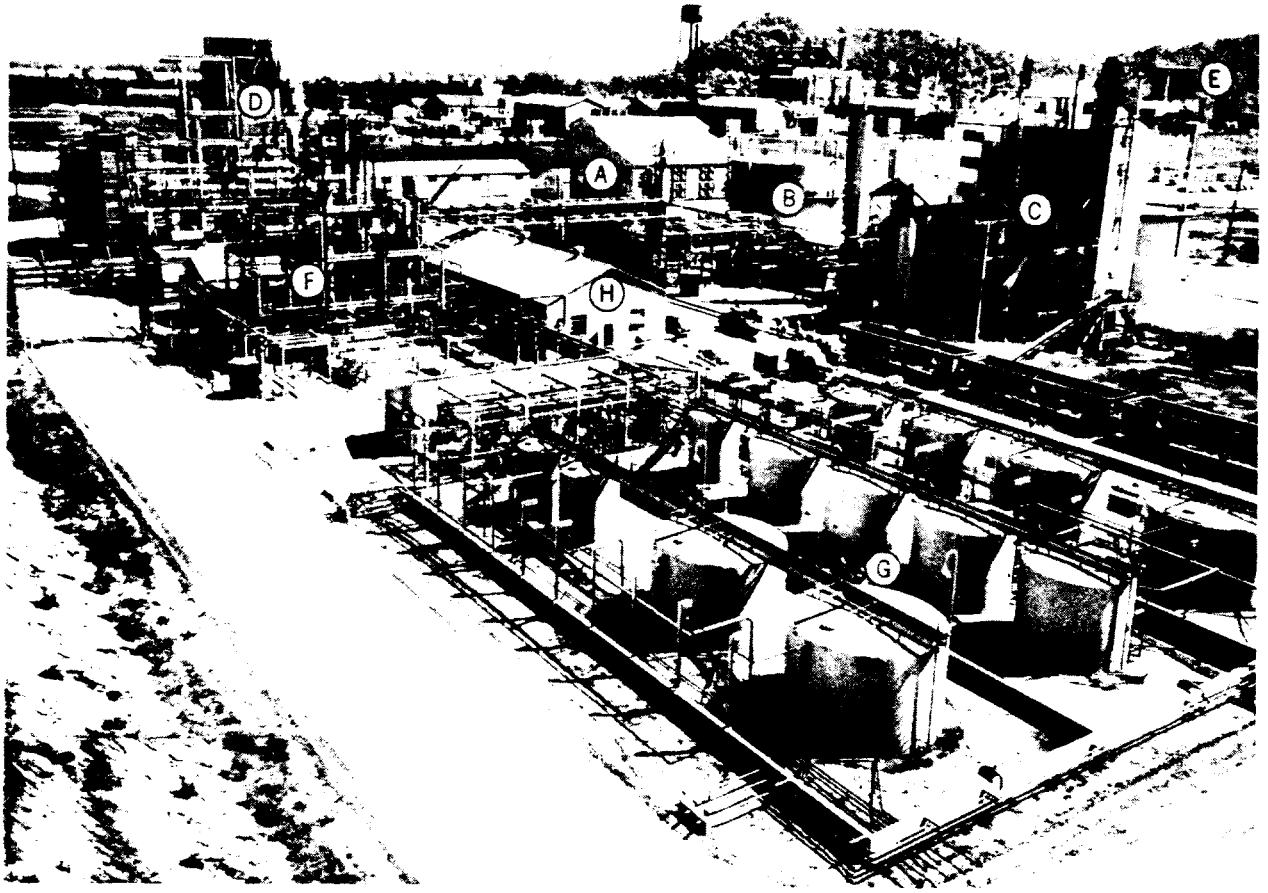

FIGURE 1. - Gas-Synthesis Demonstration Plant: A, Oxygen plant; B, oxygen holder; C, gasification and purification; D, synthesis gasholder; E, synthesis gasholder; F, gas synthesis and liquid refining; G, tank farm; H, compressor house.

than 100 grains of ash per 100 cubic feet, the gas from the washer-cooler is passed through two electrostatic precipitators for final cleanup. The clean gas is piped to a gasholder. Gas from the holder is compressed and scrubbed with diethanolamine (DEA) to remove carbon dioxide and most of the hydrogen sulfide and organic sulfur. Two ferric oxide and two activated-carbon absorbers, both pairs in series, remove the rest of the sulfur. The purified gas then enters the synthesis reactor.

Reaction products and unreacted gas from the top of the reactor pass through an exchanger and a water-cooled condenser and collect in an overflow-type separator. Almost all the oxygenated compounds formed in the synthesis settle out in a water layer.

Noncondensables from the process stream are drawn off the top of the separator, compressed, and passed into a monoethanolamine (MEA) absorber that reduces the concentration of carbon dioxide to 2 percent. Eighty-five percent

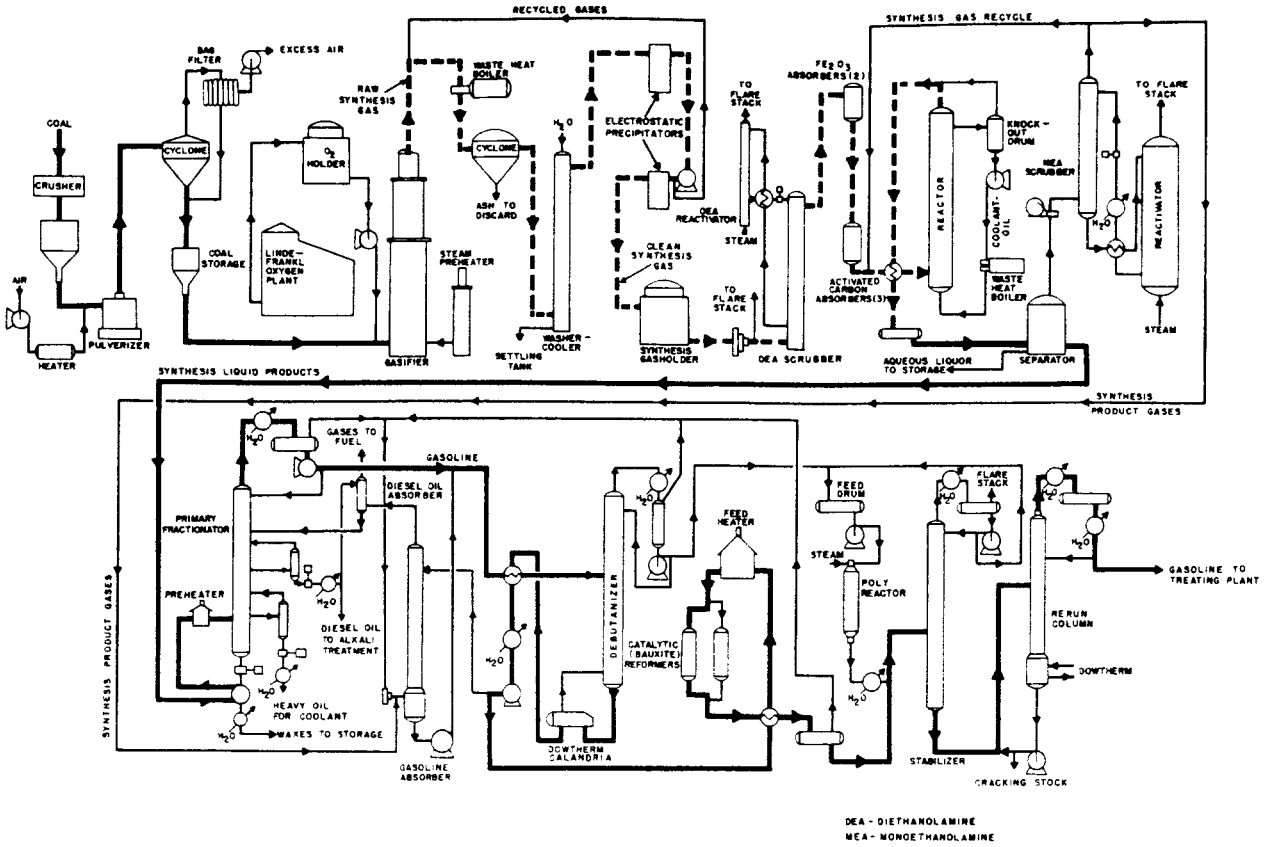

FIGURE 2. - Flowsheet of Gas-Synthesis Process.

of the carbon dioxide-free gas is recycled to the reactor; the rest goes to the distillation section where the hydrocarbons are recovered and processed.

Liquid synthesis products from the separator are transferred to the distillation area for refining. Products of refining include gasoline, diesel oil, heavy oil suitable for coolant oil or cracking stock, and paraffin-type waxes that can be cracked later.

The heavy distillate is drawn off the primary fractionator, steam-stripped of its light ends, and cooled. It is then pumped to storage, used for fuel oil or coolant oil, or is cracked and recycled.

The diesel-oil cut is stripped in an auxiliary column. About 75 percent of the oil is passed through an absorber for recovery of light ends and then is returned to the fractionator. The other 25 percent is cooled and sprayed with 10 percent caustic to neutralize and remove acids.

The overhead from the top of the primary fractionator is processed by conventional refining methods, including polymerization and catalytic reforming, to produce a gasoline.

OXYGEN PLANT

Oxygen of about 99-percent purity was produced in a 24-ton-per-day Linde-Frankl plant that functioned as required for 4 years without serious interruption. In one series of runs the plant operated continuously for 1,400 hours and was shut down only 12 hours. Because of the reliability of the plant, oxygen eventually was considered as a utility (table 2).

TABLE 2. - Typical operating data from 24-ton-per-day Linde-Frankl oxygen plant used at the Gas-Synthesis Demonstration Plant

Oxygen (98.7 percent) produced.....standard cubic feet per hour	20,625
Process air:	
To O_2 regenerators.....do.....	22,400
To N_2 regenerators.....do.....	95,200
To column[1].....do.....	11,600
Total.....do.....	129,200
Electricity per 1,000 cubic feet of O_2 produced.....kilowatts	19.2
Plant operating factor.....percent	95
Operators per shift.....	2
Oxygen:[2]	
Cost per 1,000 standard cubic foot.....	$0.80
Cost per ton.....	18.00

[1] High-pressure air (3,000 p.s.i.).

[2] Includes utilities, labor, maintenance, overhead, and burdens but not taxes, insurance, or amortization. Amortization on such a small plant over a 15-year period would be approximately $0.26/1,000 std.c.f. oxygen produced. Cost of power assumed $0.007/kw.-hr.

COAL AND COKE GASIFICATION

Horizontal Gasifier

A horizontal gasifier developed by the Koppers Co.[8] [9] was used in the first gasification trials at Louisiana. This gasifier was a 6-1/2-foot-diameter by 9-foot-long cylinder lined with silica brick and operated at atmospheric pressure. Superheated steam and a mixture of powdered coal and oxygen were fed into both ends of the cylinder, and product gases were re-removed at the middle near the top. A pebble heater superheated the process steam to between 1,500° and 2,200° F. Table 3 includes typical performance data for the horizontal gasifier.

The horizontal gasifier was not operated after the process variables had been investigated to the point where a commercial design was possible. This information was used when a commercial plant furnishing gas for ammonia

[8] Dressler, R. G., Batchelder, H. R., Tenney, R. F., Wenzell, L. P., Jr., and Hirst, L. L., Operation of a Powdered Coal Gasifier at Louisiana, Mo.: Bureau of Mines Rept. of Investigations 5038, 1954, 35 pp.

[9] Work cited in footnote 5, p. 2.

synthesis was built in Finland by the Heinrich Koppers Co., Essen, Germany. This plant operated successfully.[10] In experimental work on atmospheric-pressure gasification, the Bureau of Mines at Morgantown, W. Va., and others developed a vertical gasifier that appeared promising for use in the gas-synthesis process.[11-14] Accordingly, a vertical gasifier was constructed that was similar to a smaller unit developed at Morgantown. The work with the vertical gasifier was a necessary step in the later development of a pressure gasifier at Morgantown.[15-17]

Vertical Gasifier

The mechanical development and the performance of 15 runs with a 4-foot inside diameter by 20-foot, Morgantown-type unit has been described.[18 19] After these 15 runs, this unit was extensively revised; a coal-feeding system, in which a rotary, mechanical feeder fed powdered coal into a jet flow of the process steam was installed. A second reactant-injection burner (nozzle) that fired diametrically opposite the original single burner was also added,

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[10] Totzek, F., Synthesis Gas From the Koppers-Totzek Gasifier: Chem. Eng. Prog., vol. 50, No. 4, April 1954, pp. 182-187.

[11] Strimbeck, G. R , Holden, J. H., Rockenbach, L. P., Cordiner, J. B., Jr., and Schmidt, L. D., Pilot-Plant Gasification of Pulverized Coal With Oxygen and Highly Superheated Steam: Bureau of Mines Rept. of Investigations 4733, 1950, p. 41.

[12] Strimbeck, G. R., Cordiner, J. B., Jr., Baker, N. L., Holden, J. H., Plants, K. D., and Schmidt, L. D., Gasification of Pulverized Coal With Steam and Oxygen at Atmospheric Pressure: Bureau of Mines Rept. of Investigations 5030, 1954, 37 pp.

[13] Sebastian, J. J. S., Edeburn, P. W., Bonar, F., Bonifield, L. W., and Schmidt, L. D., Laboratory-Scale Work on Synthesis-Gas Production: Bureau of Mines Rept. of Investigations 4742, 1951, 41 pp.

[14] Strimbeck, G. R., Holden, J. H., Bonar, F., Plants, K. D., Pears, C. D., and Hirst, L. L., Gasification of Pulverized Coal at Atmospheric Pressure: Discussion of Pilot-Plant Development, Study of Process Variables, and Relative Gasification Characteristics of Coals of Different Rank: Bureau of Mines Rept. of Investigations 5559, 1960, 68 pp.

[15] McGee, J. P., Schmidt, L. D., Danko, J. A., and Pears, C. D., Pressure-Gasification Pilot Plant Designed for Pulverized Coal and Oxygen at 30 Atmospheres: AIME Symposium on Gasification and Liquefaction of Coal, New York, N. Y., Feb. 20-21, 1952, pp. 80-108.

[16] Strimbeck, G. R., Cordiner, J. B., Jr., Taylor, H. G., Plants, K. D., and Schmidt, L. D., Progress Report on Operation of Pressure-Gasification Pilot Plant Utilizing Pulverized Coal and Oxygen: Bureau of Mines Rept. of Investigations 4971, 1953, 27 pp.

[17] Holden, J. H., Strimbeck, G. R., McGee, J. P., Willmott, L. F., and Hirst, L. L., Operation of Pressure-Gasification Pilot Plant Utilizing Pulverized Coal and Oxygen. A Progress Report: Bureau of Mines Rept. of Investigations 5573, 1960, 56 pp.

[18] Work cited in footnote 5, p. 2.

[19] Batchelder, H. R., and Hirst, L. L., Coal Gasification at Louisiana, Mo.: Ind. Eng. Chem., vol. 47, No. 8, August 1955, pp. 1522-1528.

and the results of using this redesigned gasifier were reported.[20] This vertical gasifier was operated in conjunction with the gas-purification section of the demonstration plant. Table 3 shows typical results obtained with the vertical gasifier.

TABLE 3. - Typical performance of horizontal, vertical and Kerpely gasifiers

	Horizontal	Vertical	Kerpely
Feed rate:			
Coal[1].....pounds per hour	2,500	2,200	-
Coke.....do..	-	-	1,700
Oxygen.....standard cubic feet per hour	22,000	21,000	15,000
Steam.....pounds per hour	2,000	2,300	2,500
Products:			
Synthesis gas...standard cubic feet per hour	78,000	73,000	79,000
$CO + H_2$.....do..	62,000	58,000	63,000
Raw materials, per thousand standard cubic feet of $CO + H_2$ produced:			
Dry coal.....pounds	42	39	-
Dry coke.....do..	-	-	27
Oxygen.....standard cubic feet	355	370	241
Steam.....pounds	40	400	39
Gas composition:			
CO.....percent	41	42	43
H_2.....do..	38	37	37
CO_2.....do..	16	17	17
Carbon gasified.....do..	80	85	-

[1]High-volatile C bituminous, Rock Springs, Wyo.

Kerpely Producer

Synthesis gas from a Kerpely producer was used in developing the gas-purification system and in the runs with the synthesis section. This unit, a standard, 7-foot-diameter vessel, was fed with coke and a continuous stream of oxygen instead of the normally used air supply. The oxygen was premixed with steam. Results of the gasification of coke have been described.[21 22] Table 3 shows typical data. The Kerpely unit, comparatively free from operating difficulties, was a dependable source of raw gas. Thus, the major effort could be focused on the gas purification and synthesis phases of the program.

[20]Work cited in footnote 19, p. 6.

[21]Batchelder, H. R., Dressler, R. G., Tenney, R. F., Kruger, R. E., and Segur, R. D., Operation of Kerpely Producer With Oxygen-Enriched Blast at Bureau of Mines, Louisiana, Mo.: Pres. at AGA Convention, Atlantic City, N. J., October 1950, pp. 348-354.

[22]Willmott, L. F., Batchelder, H. R., and Tenney, R. F., Production Operating Experience With Oxygen in the Kerpely Producer at Louisiana, Mo.: Bureau of Mines Rept. of Investigations 5108, 1955, 16 pp.

SYNTHESIS-GAS PURIFICATION

Process Description

Raw synthesis gas was passed to a gasholder and through, successively, a waste heat boiler, a cyclone-type dust separator, a washer-cooler, and two electrostatic precipitators (fig. 2). From the gasholder, the virtually dust-free gas was flared, or sent to compression, purification, and synthesis.

In a three-step treatment, the purification system removed carbon dioxide, hydrogen sulfide, and organic sulfur. First, gas from the holder was compressed to the normal operating pressure of 350 pounds per square inch gage and scrubbed with 40 percent diethanolamine to remove most of the carbon dioxide and sulfur. (Scrubbing with diethanolamine is a conventional operation, except for the use of a 40-percent amine.) Second, the remaining hydrogen sulfide was removed in two absorbers (boxes) packed with ferric oxide on wood chips. The packing was 25 percent ferric oxide and weighed about 30 to 32 pounds per cubic foot. Because most of the hydrogen sulfide was removed in the amine scrubber, the load on the iron boxes was light, and recharging was required infrequently. Finally, the gas was passed through activated-carbon absorbers to remove organic sulfur. These absorbers, which were designed and operated in accordance with standards set by the Bureau at Morgantown, were 4 feet in diameter and were packed with carbon to a depth of 9 feet. Approximately 3 million cubic feet of gas was processed through two absorbers in series for 48 hours. During this operation a third absorber was regenerated for 10 hours or more with 600° F. superheated steam and later was placed on stream in second position. Carbon losses in the absorbers were insignificant, and activation of carbon appeared unlimited. The ability of the activated carbon to desulfurize gas to trace quantities at 350 pounds per square inch gage yet retain its activity after repeated cyclic regeneration suggests that in the future industrial gases may be treated by this method.

A fourth purification step, which was used in the first trials of the purification system, sent the gas over hot, alkalized ferric oxide; this procedure is similar to the standard two-stage treatment practiced in Germany. (The alkalized ferric oxide absorbers are not shown in figure 2.) The temperature of the first alkalized ferric oxide tower, beginning at 390° F., was raised over a 50-day period to about 500° F. At the same time the temperature in the second tower was raised from 350° to 420° F. The towers were not operated successfully, however, because it was difficult to control the temperature of the alkalized ferric oxide (gas-synthesis run 1). This difficulty undoubtedly could have been circumvented, but the fourth purification step was eliminated, owing to the efficient performance of the three preceding steps. Thus, the expense of handling the expendable ferric oxide was avoided.

Performance

Purifying Gas From Kerpely Producer

Clean gas for the gas-synthesis runs described later in this report and for all the preceding trials of the purification system was made in the

Kerpely producer. Results are described in previous publications.[23] Table 4 gives typical performance data.

TABLE 4. - Typical performance of synthesis gas purification system with gas from the Kerpely producer

(Feed, 75,000 c.f.h.; gas from DEA scrubber, 63,000 c.f.h.; amine, 38 g.p.m.; absorber, 350 p.s.i.g.)

Gas from--	CO_2, percent	H_2S, grains per 100 standard cubic feet	Organic sulfur, grains per 100 standard cubic feet
Kerpely producer.....	19	75	1.5
DEA scrubber[1].....	3	3	1
Fe_2O_3 boxes.....	-	([2])	.01
Activated-carbon absorbers.	-	([3])	[4]<.005

[1]DEA = diethanolamine. [2]Pass Pb $(C_2H_3O_2)_2$ test. [3]Trace. [4]Generally accepted tolerance for Fischer-Tropsch Catalyst, 0.1 grain per 100 std.c.f.

Purifying Gas From Vertical Gasifier

A combined gasification-purification operation was carried out with gas from the vertical gasifier after the four gas-synthesis runs were completed and before all experimental work was terminated at the Louisiana plant. This 145-hour operation was particularly significant because it was the first in which the purification system was operated with gas produced directly from coal. As indicated above, all previous purification operations had been confined to the treatment of Kerpely-derived gas, which had a much lower concentration of hydrogen sulfide. Table 5 gives typical performance with the gas from the vertical gasifier. Because (1) the DEA scrubber was clean before the run, (2) the feed rate was lower than usual, and (3) the carbon dioxide content of the gas was lower than usual, the amine rate of 43 gallons per minute probably was higher than necessary for optimum performance, as shown by the very low carbon dioxide content of the scrubbed gas. It was still apparent, despite these advantages, that the unit performed better than in any previous operation.

TABLE 5. - Performance of synthesis gas purification system with gas from vertical gasifier

(Feed, 62,000 c.f.h.; gas from DEA scrubber, 50,000 c.f.h.; amine, 43 g.p.m.; absorber, 380 p.s.i.g.)

Gas from--	CO_2, percent	H_2S, grains per 100 standard cubic feet	Organic sulfur, grains per 100 standard cubic feet
Vertical gasifier.....	18.0	600	20
DEA scrubber.....	.2	3	1.5
Fe_2O_3 absorbers.....	-	([1])	.01
Activated-carbon absorbers.	-	([2])	([2])

[1]Pass Pb $(C_2H_3O_2)_2$ test. [2]Trace.

[23]Wenzell, L. P., Jr., Dressler, R. G., and Batchelder, H. R., Plant Purification of Synthesis Gas: Ind. Eng. Chem., vol. 46, No. 5, May 1954, pp. 858-862.

Table 6 shows the effect of hydrogen sulfide in the gas from the Kerpely producer and the vertical gasifier on the rich (unregenerated) amine solution.

TABLE 6. - Performance of reactivator on diethanolamine used to treat gas from vertical gasifier or Kerpely producer[1]

Gas from--	H_2S, grains per 100 standard cubic feet	CO_2 and H_2S-rich DEA from scrubber		Lean DEA from reactivator		Acid gas, analysis, percent[2]			
		H_2S, grains per gallon	CO_2, milliliter per milliliter	H_2S, grains per gallon	CO_2, milliliter per milliliter	CO_2	H_2S	CH_4	N_2
Gasifier....	600	196	33	47	3	86.6	5.1	6.0	2.3
Kerpely.....	75	53	46	30	4	85.0	.5	10.0	4.5

[1]Operating data for gas from vertical gasifier: Feed, 62,000 std. c.f.h.; gas from DEA scrubber, 50,000 std. c.f.h.; amine rate, 43 g.p.m.; absorber, 380 p.s.i.g. Operating data for gas from Kerpely: Feed, 76,000 std. c.f.h.; gas from DEA scrubber, 64,000 std. c.f.h.; amine rate, 38 g.p.m.; absorber, 380 p.s.i.g.

[2]About 1,000 cu.ft. of natural gas fed to reactivator reboiler to inhibit foaming.

The ferric oxide absorbers operated routinely, and hydrogen sulfide always was removed completely in the first unit. Operation of the activated-carbon absorbers also was routine. Even after a newly steamed absorber was put on stream, the total sulfur at the exit of the second absorber never exceeded a trace. In all other respects the performance of the activated-carbon absorbers was much like that of the previous operations.

The foregoing results indicate that dust-free gas made directly from coal can be routinely purified under virtually the same conditions and at the same cost that gas from the Kerpely producer can be purified.

SYNTHESIS AND REFINING OF LIQUID PRODUCTS

Process Description

Liquid products were synthesized in a 3-foot-inside-diameter by 30-foot-high reactor operated at about 300 pounds per square inch gage and 450° to 525° F. (figs. 3 and 4). Approximately 7 tons (116 cubic feet) of synthetic-ammonia-type fused-iron catalyst[24] was charged onto a bed of 3/8-inch- to 3-inch-diameter iron spheres in the bottom of the reactor. This quantity of catalyst permitted a space velocity of about 535 volume per volume per hour with a flow of 67,000 standard cubic feet per hour of purified synthesis gas. The iron spheres distributed the gas evenly and supported the catalyst above the gas inlet.

[24]Kastens, M. L., Hirst, L. L., and Dressler, R. G., An American Fischer-Tropsch Plant: Ind. Eng. Chem., vol. 44, No. 3, March 1952, p. 460.

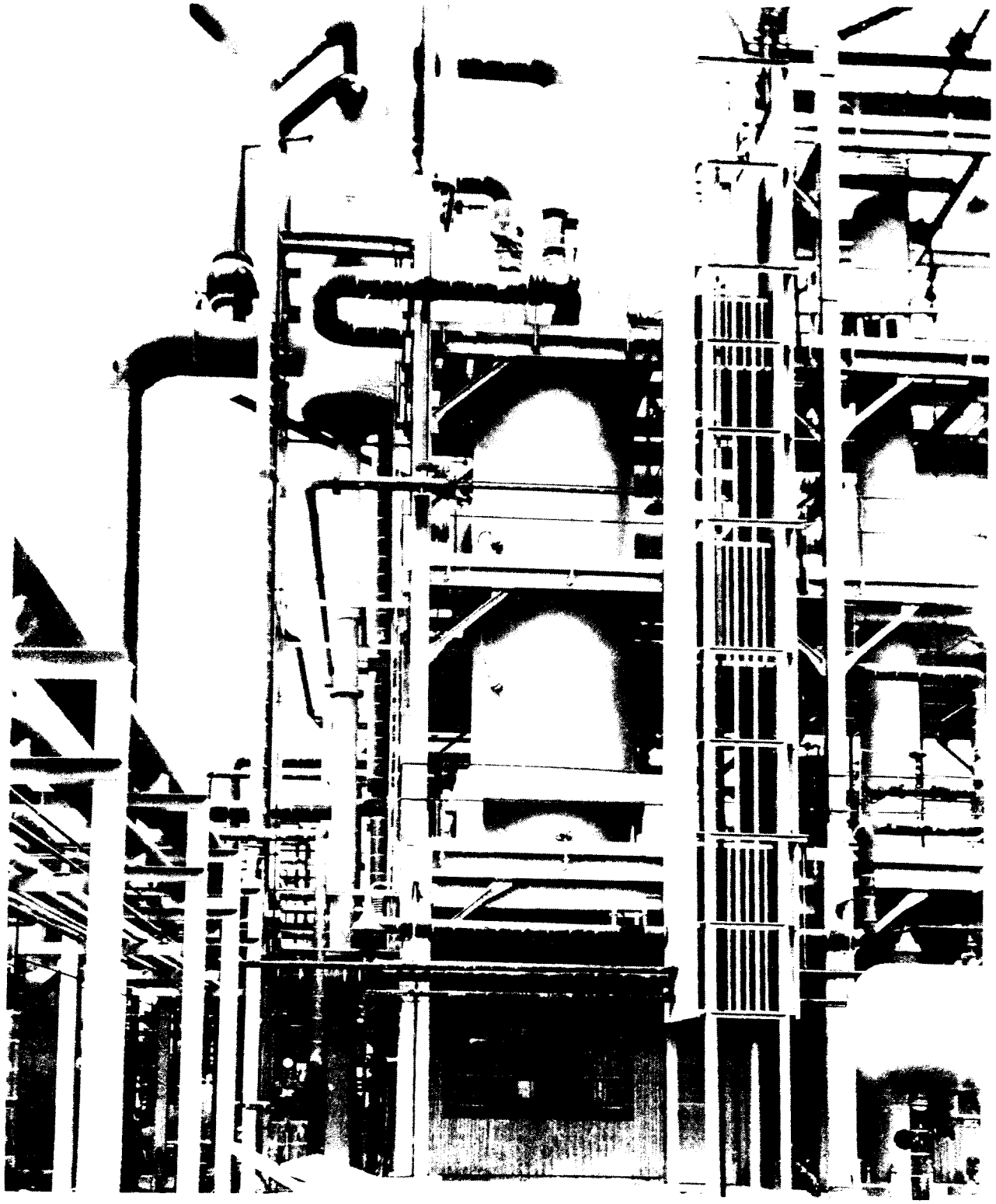

FIGURE 3. - Reactor for Conversion of Synthesis Gas to Liquid Hyarocarbons.

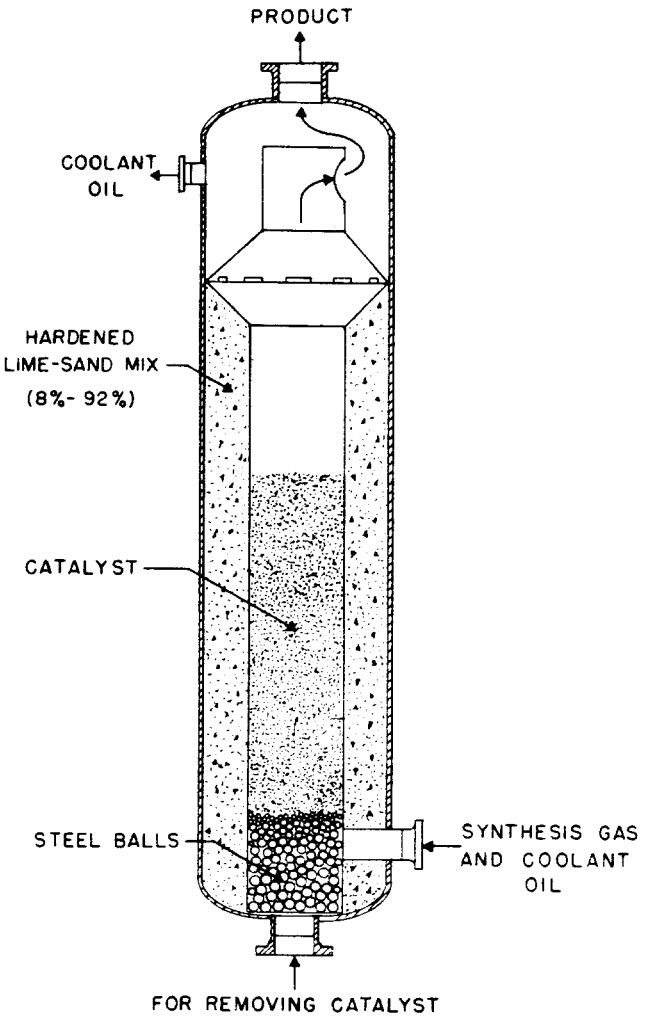

FIGURE 4. - Synthesis Reactor.

Purified synthesis gas and coolant oil were pumped into the reactor, where the gas came into intimate contact with the catalyst, which was kept in semisuspension by the upward flow of coolant oil. The heated coolant oil from the top of the reactor passed to a knockout drum that separated volatile compounds and to a waste-heat boiler that generated steam at 275 pounds per square inch gage. After exchanging heat with the incoming synthesis gas, the gas was scrubbed; the major part was recycled to the reactor. The products were transferred to other units for processing. Figure 5 shows the distillation section.

Performance

Four runs with the complete demonstration plant (including the oxygen plant, Kerpely producer, gas purification units, and synthesis and refining sections) were continued for 8, 25, 21, and 39 days, respectively.

Run 1

This 8-day run served to train the operators, demonstrate the operability of the entire process, and point the way to improvements. Process studies gave pertinent information on the effectiveness of each intermediate step. During the induction of the fused-iron catalyst the temperature of the reactor was gradually raised to 450° F. Then the temperature was raised in intervals of 25° F.

Some reaction was observed at 500° F., but even when the temperature was elevated to 550° F. the conversion was only 30 to 32 percent. The beginning coolant-oil flow was 250 gallons per minute, and the fresh gas feed was 60,000 standard cubic feet per hour. A recycle-gas flow of 60,000 standard cubic feet per hour was maintained at first, but this rate had to be reduced, owing to excessive entrainment of coolant oil in the gas stream. Operating pressure was 325 to 330 pounds per square inch gage throughout. Enough catalyst was used in the reactor to permit a space velocity of 600 volume per volume per hour based on the anticipated flow of fresh gas. Data from run 1 are given in table 7. (Note: Units in this report usually are given in the English system. Occasionally, as in table 7, they are given in the metric system for consistency with units normally used by other investigators of synthesis technology.)

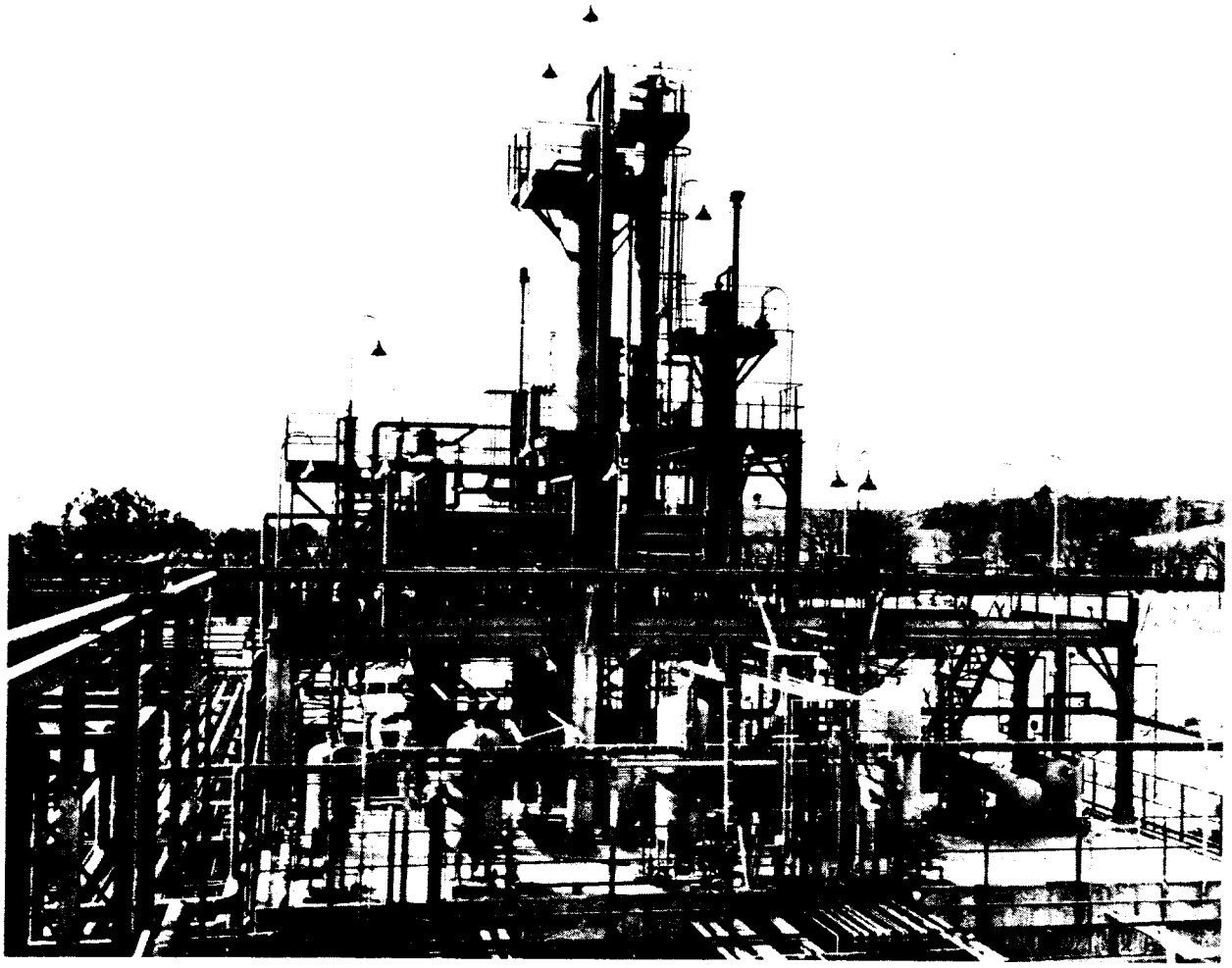

FIGURE 5. - Distillation Unit for Refining Liquid Hydrocarbons From the Synthesis Reactor.

Run 2

Before the run the Kerpely producer was repaired and the piping in the gas-purification system was changed to bypass the alkalized ferric oxide towers. In the synthesis and distillation units, the fused-iron catalyst was replaced; many piping changes were made; valves were replaced; instruments were readjusted; and various controls were put in working order.

The catalyst was inducted in a stepwise procedure. This operation covered 5 days; the beginning temperature of 430° F. was raised about 25° F. per day as in run 1. The conversion of synthesis gas increased from 14 to 72 percent during the induction of catalyst. After the induction, temperatures were maintained between 522° and 528° F. Approximately 60,000 standard cubic feet per hour of feed gas was passed into the reactor, with the recycle rate eventually reaching 90,000 standard cubic feet per hour. At first, coolant oil was circulated at 300 gallons per minute, but in the later stages of the induction phase the flow was increased to 500 gallons per minute, where

it was maintained during the synthesis operation. Table 7 shows characteristic operating data and results from run 2.

TABLE 7. - Operating conditions for runs 1 and 2 with the integrated Gas-Synthesis Demonstration Plant

	Run 1	Run 2
Fresh gas to reactor.....standard cubic feet per hour	56,300	62,600
H_2 to CO ratio in fresh gas to reactor.....	.82	.80
Recycle ratio (gas).....	([1])	1.55
Maximum temperature in reactor.....degrees Fahrenheit	549	523
Temperature drop across reactor.....do..	9	17
Maximum pressure in reactor....pounds per square inch gage	330	329
Pressure drop across reactor.....do..	22	31
Space velocity..... volume per volume per hour	-	537
Conversion of CO + H_2.....percent	31	81
Usage ratio, H_2 to CO.....	.68	.71
C_1 + C_2 yield[2] per cubic meter CO + H_2 in fresh feed.grams	17.5	34.2
C_3+ yield[2].....do..	63.7	133.4
CO_2:		
In fresh gas to reactor.....percent	1.6	1.6
In recycle and tail gases from absorbers.....do..	.1	.1
In gas from reactor.....do..	11.8	11.5

[1]No recycle.
[2]Theoretical.

Except for processing the gasoline in a Perco catalytic-reforming unit (accomplished later), all units of the distillation system were operated during some part of the run. (In the Perco process the gasoline is passed over heated bauxite to improve the octane rating by isomerization and by removal of oxygenated compounds.) During the first days of operation, the primary fractionator was used to remix and recirculate the products. The absorbers and debutanizer also were placed in service. After being put on stream, the polymerization unit began to function within 2 days.

Approximately 15,000 gallons of synthetic oil was recovered. This was only partial recovery of products, as little or none of the C_3 and C_4 hydrocarbons were recovered and converted to polymer gasoline. Product distribution in gallons for run 2 was as follows: Raw gasoline, 11,150; diesel oil, 790; cracking stock, 1,010; bottoms, 1,720. Approximately 8,950 gallons of the raw gasoline was given the bauxite treatment, that is, reformed to improve its octane rating. Because most of this run was made at outdoor temperatures down to 0° F., preheat temperatures 200° to 300° F. above normal were required to offset heat losses in the lines. Table 8 gives data on the reformed gasoline. Owing to the low temperatures in the catalyst chamber, the clear octane improvement of only 8.7 and 7.5 numbers was not as great as anticipated. As can be seen, the addition of 1 and 3 milliliters of tetraethyllead per gallon raised the octane numbers to 80.0 and 86.4 and to 75.1 and 78.6 for the research and motor method values, respectively.

TABLE 8. - Properties of synthetic gasoline produced in run 2 at the Gas-Synthesis Demonstration Plant

	Raw	Reformed
Gasoline.....gallons	8,950	7,200
Gravity, at 60° F.....° API	66.5	67.5
Reid vapor pressure.....pounds per square inch gage	7.6	6.8
Distillation, ASTM:		
Initial boiling point.....degrees Fahrenheit	91	97
5 percent.....do...	112	116
10 percent.....do...	126	133
20 percent.....do...	150	156
30 percent.....do...	175	178
40 percent.....do...	199	201
50 percent.....do...	224	226
60 percent.....do...	249	251
70 percent.....do...	273	277
80 percent.....do...	297	306
90 percent.....do...	335	345
95 percent.....do...	369	371
End point.....do...	407	400
Octane ratings:		
Research method (ASTM D908):		
Clear.....	61.5	70.2
+1 milliliter TEL per gallon[1].....	72.0	80.0
+3 milliliters TEL per gallon.....	82.6	86.4
Motor method (ASTM D357):		
Clear.....	57.7	65.2
+1 milliliter TEL per gallon.....	68.5	75.1
+3 milliliters TEL per gallon.....	75.2	78.6

[1]TEL = tetraethyl lead.

The diesel oil was washed with caustic soda and given a weathering treatment with steam to adjust the flashpoint. Commercial laboratory tests showed that the oil met the military specifications of Class 1 diesel oil, MIL-D-896 (table 9).

Some of the sulfur in the product may have been present in the synthesis gas, but the sulfur probably came from the sealing oil used in the coolant-oil circulating pumps.

Run 3

The third run was interrupted after about 2 weeks by repeated failure of the coolant-oil circulation pumps. Abrasion of the pumps by disintegrated catalyst was responsible for much of the difficulty. Operations were resumed about 1 week later, and the run was terminated after 21 days of operation. The oxygen plant, Kerpely producer, and gas-purification section performed satisfactorily.

TABLE 9. - Characteristics of synthetic diesel oil produced in run 2 at the Gas-Synthesis Demonstration Plant

	Product	Specification
Cetane rating	60	50
Flash point, degress Fahrenheit	196	[1]140
Pour point, do	-5	[2]0
Kinematic viscosity at 100° F., centistokes	2.44	2.1-6.0
Carbon residue on 10 percent residium, percent	.06	[2].20
Sulfur, do	.02	[2]1.25
Color, NPA	1.75	[2]5
Corrosion (3 hours at 212° F.)	([3])	([4])
Gravity, at 60° F., ° API	43.8	-
Distillation, ASTM:		
Initial boiling point, degrees Fahrenheit	298	-
5 percent, do	458	-
10 percent, do	462	-
20 percent, do	470	-
50 percent, do	490	-
70 percent, do	504	-
90 percent, do	528	[2]675°
End point, do	578	[2]725°

[1]Minimum.
[2]Maximum.
[3]Negative.
[4]Pass.

Conversion of CO + H_2 was 65 to 80 percent at 535° F. and 330 pounds per square inch gage. The space velocity of the purified synthesis gas approached 600 volume per volume per hour early in the run, but later was 350 volume per volume per hour, corresponding to feed rates of 68,000 and 40,000 standard cubic feet per hour, respectively.

Approximately 7,000 gallons of debutanized gasoline was reformed. This operation was carried out at a pressure of 40 pounds per square inch gage and a feed rate of 2.2 gallons per minute, which corresponds to a liquid space velocity of 1.17 volume per volume per hour. The reforming unit performed satisfactorily at 700°, 750°, 775°, and 825° F. Table 10 compares the feed stock and the finished products. The improvement in octane was approximately the same as that of the treated gasoline from the second run, but the octane rating was lower. The quality of gasoline, before and after treatment, was inferior to that of the gasolines produced in the pilot-scale work at Bruceton and the bauxite-treated gasoline synthesized by others in fluidized-bed Fischer-Tropsch operations with iron catalysts.[25] This may be attributed to the loss of some of the light ends and to operating conditions that produced a more highly saturated product.

[25]Helmers, C. J., Clark, A., and Alden, R. C., Catalytic Treatment of Synthetic Gasoline: Oil Gas Jour., vol. 47, No. 26, Oct. 28, 1948, pp. 86-92.

TABLE 10. - Properties of raw and bauxite-treated gasoline produced in run 3 at the Gas-Synthesis Demonstration Plant

	Raw	Reformed
Gravity at 60° F.....°API	65.4	66.8
Reid vapor pressure.....pounds per square inch gage	5.7	7.6
Distillation, ASTM:		
Initial boiling point.....degrees Fahrenheit	118	110
20 percent.....do...	178	173
50 percent.....do...	238	232
70 percent.....do...	284	274
End point.....do...	440	390
Octane ratings (clear):		
Research method (ASTM D908):.....	57.6	-
Bauxite-treated at-		
700° F.....	-	69.4
750° F.....	-	70.4
775° F.....	-	70.7
825° F.....	-	71.0
Motor method (ASTM D357):.....	55.3	-
Bauxite-treated at-		
700° F.....	-	63.2
750° F.....	-	63.2
775° F.....	-	63.3
825° F.....	-	63.4
Corrosion.....	([1])	([1])
Gum (air jet).....milligrams per 100 milliliters	421.0	8.6
Acid number.....	.3	.1
Carbonyl number.....	7.1	1.0
Hydroxyl number.....	20.9	2.6

[1] Pass.

Run 4

Some of the major difficulties with the synthesis unit during the preceding runs involved (1) the coolant-oil circulating pumps, (2) the introduction of gas to the synthesis reactor, and (3) the distribution of the gas in the reactor. Therefore, several design changes were made in an attempt to eliminate these troubles before run 4 was begun. First, the piping to the seals of the three coolant-oil circulating pumps was modified to provide for a separate supply of oil. Also, a new method was devised for introducing coolant oil and synthesis gas to the reactor. The latter change, which involved the use of a scalloped bell as the gas inlet and eliminated the steel-ball packing in the bottom of the reactor, was designed to give a more uniform gas flow and reduce the carryover of catalyst. Figure 6 shows the redesigned unit.

Another major difficulty in runs 2 and 3, which were conducted at 525° and 535° F., respectively, was disintegration of the catalyst. In run 4, however, operating at 515° F. was expected to help maintain the physical integrity of the catalyst.

Approximately 13,700 pounds of the synthetic-ammonia-type fused-iron catalyst was inducted over a 5-day period at temperatures of 400°, 460°, 480°, 500°, and 515° F. During the induction and most of the run the base operating conditions were as follows:

Base operating factors:		Units
Fresh feed[1]	cubic feet per hour	60,000
Tail gas	standard cubic feet per hour	10,000
Recycle gas	do..	60,000
Coolant oil	gallons per minute	500
Pressure	pounds per square inch gage	300
Space velocity	volume per volume per hour	600

[1] Maximum.

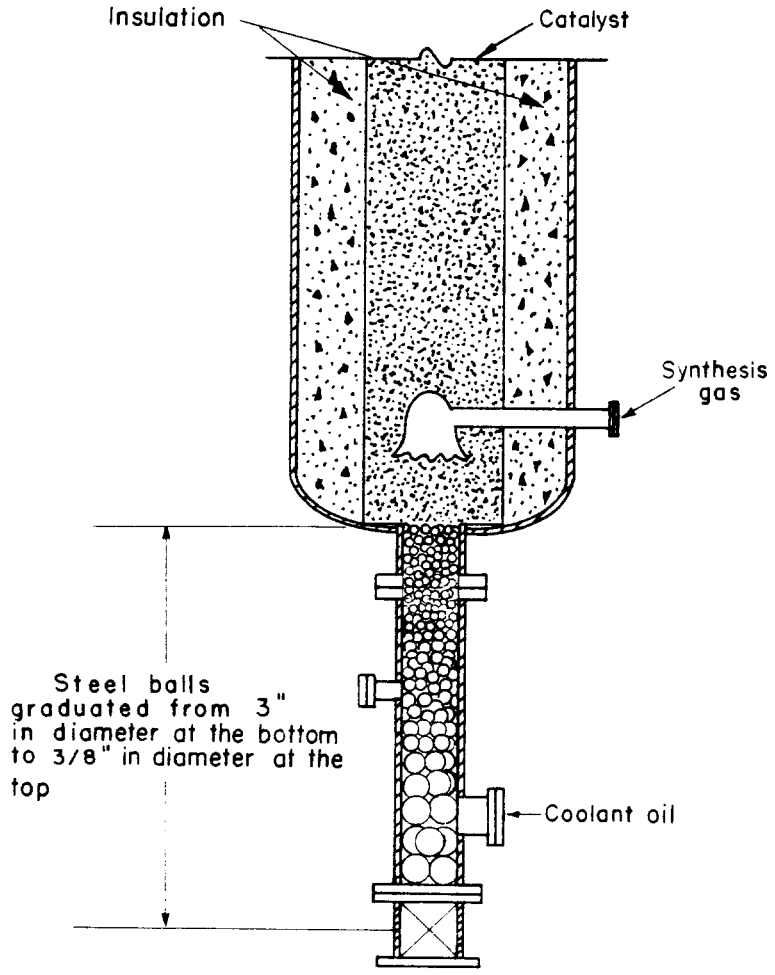

FIGURE 6. - Modified Synthesis Reactor.

Run 4 continued for 39 days, usually at the base conditions, but occasionally at space velocities of 480 and 340 volume per volume per hour. The catalyst slowly disintegrated during the run; the solids in the coolant oil (determined as ash) increased from 2 to about 25 percent, which represented about 40 percent of the original ferric oxide catalyst. The catalyst became more active however, as the percentage of finely divided catalyst in the coolant oil increased, apparently because of the additional surface made available. The mechanical-type carbon seals on the coolant-oil circulating pumps gave very little trouble, and the leakage of oil from the seals into the system was slight. The relatively few seal failures were caused by abrasion of the faces of the seals after relatively long periods of operation. The run was terminated by a leak in the housing of the raw synthesis

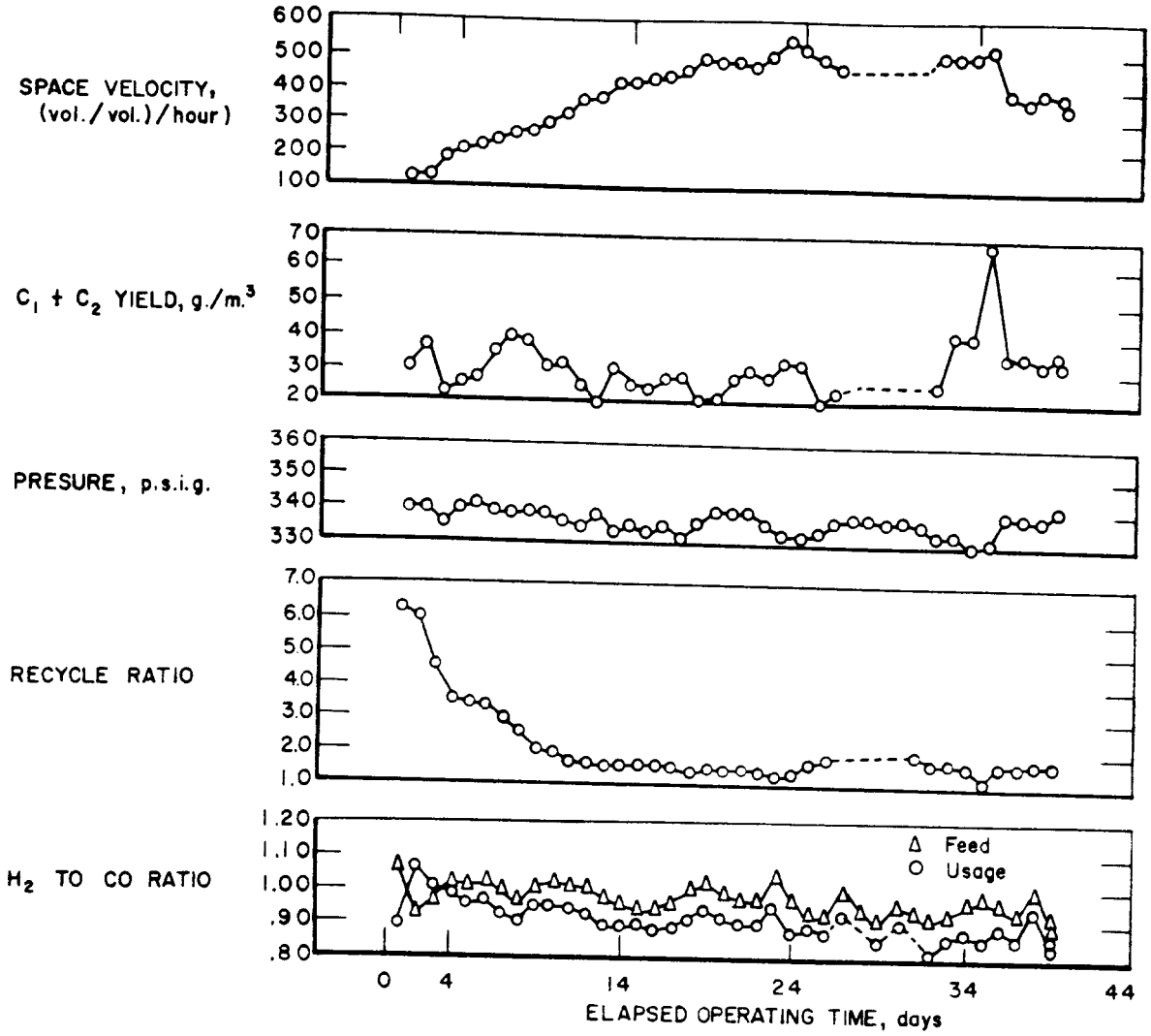

FIGURE 7. - Log of Synthesis Operation in Run 4 at the Gas-Synthesis Demonstration Plant.

gas exhauster, similar to a leak that was patched earlier. The second leak was not patched, however, because the major objectives of the run had already been attained. Figures 7 and 8 are a daily log of the synthesis operations.

When the primary recovery system was revised for run 4 (fig. 9), the primary fractionator was converted for use as a stripper. The feed heater for the fractionator was converted to a reboiler, and one of the side stream-strippers was converted to a flash tower in which the nonvaporizable part of the rich oil was separated for use in synthesis as coolant oil. Topped synthesis condensate from the stripper, having characteristics similar to a diesel oil, served as lean oil feed to the absorber. The stripper operated at low pressure, 20 pounds per square inch gage, and effectively stripped the liquid synthesis products. The stripped product contained large quantities of propane and butane that could not be condensed in the overhead condenser. These products were condensed in the wet-gas compressor aftercooler (not shown in fig. 9), were separated and were sent to the debutanizer.

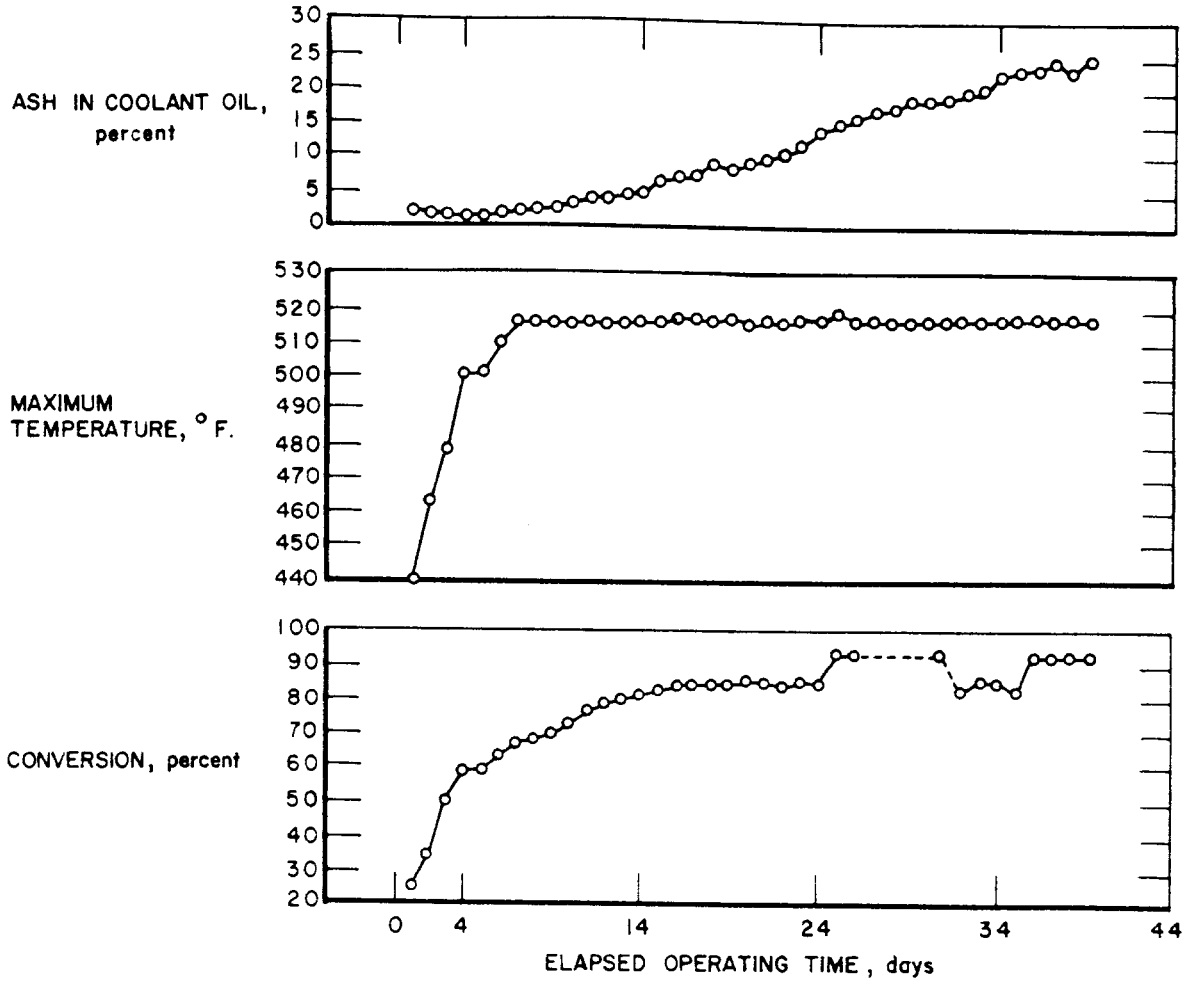

FIGURE 8. - Log of Synthesis Operation in Run 4 at the Gas-Synthesis Demonstration Plant (Con.)

The gasoline produced in run 4 was reformed, blended with polymer gasoline and butane, stabilized, and rerun to produce a finished gasoline that had an 8-pound-per-square-inch gage Reid vapor pressure and a 375° F. end point. The properties of this gasoline are shown in table 11.

Table 12 shows product distributions for operating periods in which the conversion of synthesis gas to liquid products was 84 and 92 percent. A comparison of data from the two periods shows that the yield of propane, butane, and gasoline was greater at the higher conversion level, but the actual yield of polymer gasoline was lower. A lower percentage of unsaturated compounds in the C_3 to C_4 product would account for this lower yield of polymer gasoline. This gas-synthesis process produced a lower yield of chemicals than in other synthetic liquid fuels processes. For example, the yield of oxygenated chemicals, normally 5 to 7 percent of the C_3 + products in the gas-synthesis

process, amounted to 25 percent in the fluidized process.[26] Table 13 gives the distribution of organic compounds in the aqueous condensate.

TABLE 11. - Properties of finished gasoline produced in run 4 at the Gas-Synthesis Demonstration Plant

Gravity, at 60° F.....°API	68.5
Reid vapor pressure.....pounds per square inch gage	8.9
Distillation, ASTM:	
Initial boiling point.....degrees Fahrenheit	108
5 percent.....do...	124
10 percent.....do...	136
20 percent.....do...	154
30 percent.....do...	174
40 percent.....do...	194
50 percent.....do...	212
60 percent.....do...	234
70 percent.....do...	256
80 percent.....do...	280
90 percent.....do...	320
95 percent.....do...	342
End point.....do...	362
Octane ratings:	
Research method (ASTM D908):	
Clear.....	79.2
1 milliliter TEL per gallon.....	87.2
3 milliliters TEL per gallon.....	90.8
Motor method (ASTM D357):	
Clear.....	70.3
1 milliliter TEL per gallon.....	78.1
3 milliliters TEL per gallon.....	82.0
Corrosion test (ASTM D130), 3 hours at 122° F.....	(1)
Gum:	
Preformed.....milligrams per 100 milliliters	1.0
Potential.....do...	3.2
Sulfur.....percent	.02
Induction period.....minutes	[2]240
Color, Saybolt.....	+22
Odor.....	(3)
Doctor test.....	(1)

[1] Negative.
[2] Plus.
[3] Normal.

[26] Eliot, T. Q., Goddin, C. S., Jr., and Pace, B. S., Chemicals From Hydrocarbon Synthesis: Chem. Eng. Prog., vol. 45, No. 8, August 1949, pp. 532-536.

TABLE 12. - Distribution of products from the conversion of 100,000 standard cubic feet of CO + H_2 in run 4 at the Gas-Synthesis Demonstration Plant

Operating conditions	5-day run
Maximum temperature of reactor.....° F.	515
Pressure.....pounds per square inch gage	337
Space velocity.....volume per volume per hour	480
Recycle ratio.....volume per volume	1.35
Catalyst in coolant oil, as ash.....percent	7
Usage ratio, H_2 to CO.....	0.91
Conversion.....percent	84

Products	Weight per gallon, pounds	Gasoline, gallons	Gas and oxygenates, gallons	C_3+ products, gallons	Gasoline, volume-percent	C_3+ products volume-percent	Liquid products, volume-percent	CO + H_2 converted per barrel, thousand standard cubic feet	Gasoline per cubic meter CO + H_2 converted, grams	C_3+ products per cubic meter CO + H_2 converted, grams
Carbon dioxide.....	-	-	[1]20	-	-	-	-	-	-	-
Gas (CH_4, C_2H_6).....	-	-	[1]2.9	-	-	-	-	-	-	-
Oxygenated organic compounds.....	-	-	43	-	-	-	-	-	-	-
Propane.....	4.3	-	-	30	-	20	-	140	-	21
Butane.....	4.9	13	-	-	13	-	-	326	10	-
Polymer gasoline.....	6.0	29	-	-	30	-	-	145	28	-
C_5+ 400 endpoint.....	5.8	55	-	-	57	-	-	76	51	-
Total gasoline.....	-	-	-	97	100	63	79	43	-	89
Diesel oil.....	6.6	-	-	12	-	8	10	350	-	13
Coolant oil.....	7.2	-	-	13	-	9	11	326	-	15
Total.....	-	-	-	152	-	100	100	27.5	-	138

Operating conditions	3-day run
Maximum temperature of reactor.....° F.	515
Pressure.....pounds per square inch gage	338
Space velocity.....volume per volume per hour	373
Recycle ratio.....volume per volume	1.43
Catalyst in coolant oil, as ash.....percent	24
Usage ratio, H_2 to CO.....	0.88
Conversion.....percent	92

Products	Weight per gallon, pounds	Gasoline, gallons	Gas and oxygenates, gallons	C_3+ products, gallons	Gasoline, volume-percent	C_3+ products, volume-percent	Liquid products, volume-percent	CO + H_2 converted per barrel, thousand standard cubic feet	Gasoline per cubic meter CO + H_2 converted, grams	C_3+ products per cubic meter CO + H_2 converted, grams
Carbon dioxide.....	-	-	[1]22	-	-	-	-	-	-	-
Gas (CH_4, C_2H_6).....	-	-	[1]3.6	-	-	-	-	-	-	-
Oxygenated organic compounds.....	-	-	43	-	-	-	-	-	-	-
Propane.....	4.3	-	-	35	-	22	-	120	-	24
Butane.....	4.8	18	-	-	16	-	-	233	14	-
Polymer gasoline.....	6.0	28	-	-	25	-	-	150	27	-
C_5+ 400 endpoint.....	5.8	66	-	-	59	-	-	64	61	-
Total gasoline.....	-	-	-	112	100	71	91	38	-	102
Diesel oil.....	6.6	-	-	8	-	5	7	525	-	8
Coolant oil.....	7.2	-	-	3	-	2	2	1,400	-	3
Total.....	-	-	-	158	-	100	100	26.6	-	137

[1] M std. c. f.

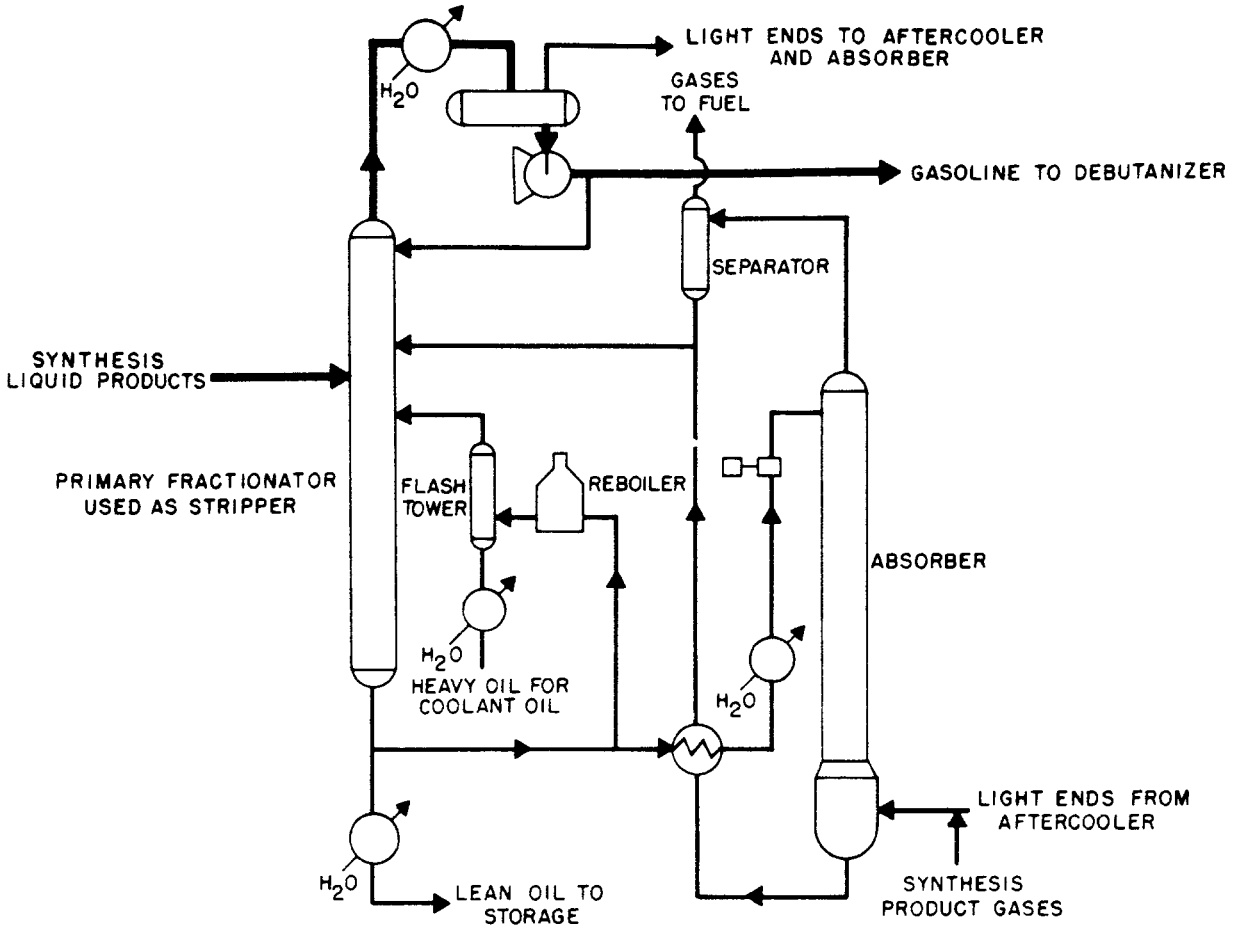

FIGURE 9. - Revised Primary Recovery Section.

TABLE 13. - Analysis of aqueous condensate produced during synthesis run 4 at the Gas-Synthesis Demonstration Plant

Component	Mole-percent[1]	Weight-percent	Volume-percent
n Butanol.....	0.6	2.1	2.5
n Propanol.....	2.2	6.3	7.5
Ethanol.....	4.6	10.1	12.2
Methanol.....	1.8	2.8	3.2
Water.....	90.6	78.1	74.0
Acetone.....	.1	.3	.3
Acetic acid.....	.1	.3	.3
Total.....	100.0	100.0	100.0

[1]Mass spectrometer analysis.

DISCUSSION

Results obtained at the demonstration plant were different in several respects from the results of the pilot-scale work at Bruceton. Although

total yields were comparable, distribution of products under like conditions was different (table 14). Because less propylene was made, the yield of polymer gasoline was lower in the pilot-scale work.

TABLE 14. - Distribution of C_3+ products made in the pilot plant at Bruceton and the Gas-Synthesis Demonstration Plant

1:1 ratio of H_2 to CO in the feed gas at 515° F.

	Pilot plant		Demonstration plant (run 4)			
	[1]0.62		[1]1.0		[1]2.71	
Distribution of C_3+	Weight-percent	Volume-percent	Weight-percent	Volume-percent	Weight-percent	Volume-percent
Propane.....percent	6.9	9.7	15.0	19.7	17.4	22.1
Total gasoline.....do.	56.5	58.0	64.5	63.8	74.0	70.9
Total heavier than gasoline.do.	36.6	32.3	20.5	16.5	8.6	7.0

[1]Ratio of saturated C_3 hydrocarbons to olefinic C_3 hydrocarbons, volume per volume.

The bauxite treatment also produced different results (table 15). A maximum octane improvement of 8 numbers was achieved in the demonstration plant; an improvement of 16 numbers was achieved in the laboratory tests at Bruceton.

TABLE 15. - Results of reforming, at Louisiana, Mo., and Bruceton, Pa., of raw gasoline produced in run 3 at the Gas-Synthesis Demonstration Plant[1]

Louisiana		Bruceton	
Reforming temperature, ° F.	Motor octane	Reforming temperature, ° F.	Motor octane
700	63.2	714	68.6
750	63.2	750	71.2
775	63.3	-	-
825	63.4	819	71.0

[1]Motor octane of raw gasoline: 55.

Development work with the gas-synthesis plant at Louisiana demonstrated the feasibility of the coal-to-liquid fuels process. The synthesis reactor was designed for a "jiggling" catalyst bed operation, in which only the coolant oil was circulated. In the later part of the runs, however, substantial amounts of catalyst had disintegrated. Nevertheless, the still-active catalyst circulated as an oil slurry, and synthesis reactions continued. Disintegration of the catalyst was overcome in the pilot-scale work but remains as the major unsolved problem at the demonstration plant. Most of the mechanical difficulties were eliminated, however, and a commercial unit probably could be designed and operated on the basis of the information obtained at Bruceton and Louisiana.

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