SYNTHETIC LIQUID FUELS

ANNUAL REPORT OF THE SECRETARY OF THE INTERIOR FOR 1951

PART I. - OIL FROM COAL

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UNITED STATES DEPARTMENT OF THE INTERIOR
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BUREAU OF MINES
J. J. Forbes, Director

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Part 1. - Oil from Coal

PREFACE

This report is submitted in accordance with the provisions of the Synthetic Liquid Fuels Act of April 5, 1944 (30 U.S.C. 321-325, as amended), which require that: "The Secretary of the Interior shall render to Congress on or before the first day of January of each year a report on all operations under this Act."

Owing to the broad scope of the content and the diversity of interests represented, the Annual Report for 1951 has been divided into two separate publications. Each has been published by the Bureau of Mines as a Report of Investigations, and the respective titles follow:

R. I. 4865, Part I - Oil from Coal.

R. I. 4866, Part II - Oil from Oil Shale.

Identical in each report, the Introduction summarizes research progress made in 1951 under the entire Synthetic Liquid Fuels program.

A free copy of these publications may be obtained by a written request to the Bureau of Mines, Publications Distribution Section, 4800 Forbes Street, Pittsburgh 13, Pa. The R. I. number and title of the publication desired should be indicated.

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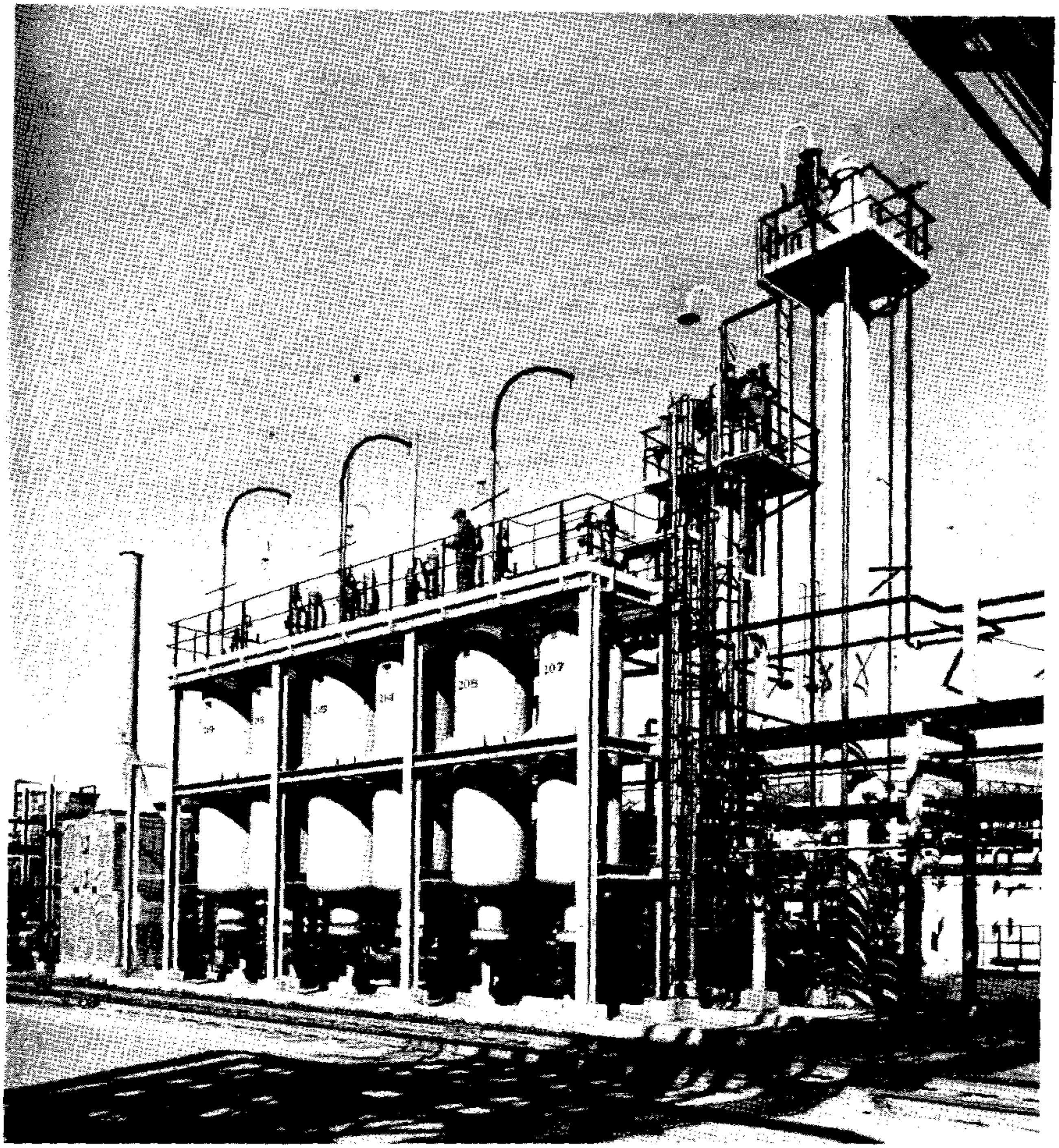

Figure 1. - Gas-purification unit of new Gas-Synthesis Demonstration Plant, Louisiana, Mo. Here harmful sulfur compounds and carbon dioxide, obtained in gasifying raw coal, are removed from compressed synthesis gas before it is fed to reactor for conversion into synthetic oil.

INTRODUCTION

Increasing demands for liquid fuels and the critical state of world affairs emphasize the importance of the Bureau of Mines synthetic liquid fuels program.

Before World War II, the United States produced all the oil necessary to meet domestic demands and, in addition, had a comfortable excess production that was used to supply a large portion of the world's needs. Soon after the war, however, domestic demands increased sharply, placing a severe burden on America's oil resources. No longer was this country able to export large quantities of oil; in fact, foreign oil was imported in increasing quantities. In spite of an increase in exploration and drilling activities, imports continued to rise; and last year, over 10 percent of the total demand was met with foreign oil.

There is strong evidence that dependence on foreign oil will continue to increase and sooner or later may create a security problem. Although it is impossible to prove that this will occur, the mere possibility of a security problem with a material as essential as oil makes it imperative to develop all possible new sources of domestic oil.

The fact that the United States is now in a position to design and construct pioneer synthetic fuels plants is a tribute to the cooperative efforts of American industry and Government. It will be remembered that in 1944, when the Bureau of Mines synthetic liquid fuels program got underway, there was no production of synthetic fuels in the United States, and the technical information available was meager indeed.

As a result of the Bureau of Mines program and the increased activity of private industry, significant progress has been made during the past 7 years. One commercial synthetic fuels plant using natural gas has been constructed; oil-shale processes are nearing commercial exploitation; coal hydrogenation has been demonstrated with American-built equipment and commercial plants have been proposed; in 1951 the Bureau successfully demonstrated a new gas-synthesis process; and significant advances have been made in developing direct coal-gasification processes.

The Bureau of Mines and the National Petroleum Council have cooperated in making studies of operating costs for coal hydrogenation and oil-shale retorting. Both the Bureau and industry have benefited by the results of this cooperative effort. The Bureau and the N.P.C. have been in good agreement on many points in these cost estimates. On some items there has been wide divergence, and it is believed advisable to continue the studies.

Oil Shale Ready for Exploitation

Some of the most outstanding achievements have been in the oil-shale phase of the program. While initially, oil shale seemingly offered only a slight promise of success, today, it generally is considered to be the most promising large-scale source of synthetic liquid fuels.

Progress in Shale Technology

Great strides have been made in all phases of the oil-shale development program, from mining the shale and extracting the crude oil to refining the oil into finished products.

One of the important developments has been the demonstration of practical methods for mining oil shale underground at the very low cost of approximately 50 cents per ton. When this cost figure was set as a goal at the outset of the mining research program, few technical experts expected that it would be reached. It was reached, however, despite an inflationary trend that has nearly doubled costs of labor and material since the work was started.

Considerable progress also has been made in retorting shale. Only 7 years ago no economical retorting process was known that was suitable for American shales. It was a question of determining the fundamental principles involved and learning how best to apply them. Today, two economical retorting processes are available. Both have been developed through the pilot-plant stage, one by the Bureau of Mines and one by the petroleum industry. Each retort is a rugged, one-vessel unit tailored to fit the particular conditions of the oil-shale regions. Neither process requires cooling water, and both provide highly efficient, continuous operation.

At the start of the synthetic fuels program, refining the crude shale oil posed numerous problems, some of which appeared to be almost insurmountable. The general opinion was that high-grade products could be made from crude shale oil only under the most severe operating conditions and at very high costs. However, concerted development efforts since have produced several suitable refining processes which yield high-grade products and yet operate at moderate conditions and reasonable cost.

Shale-oil products are used regularly in the Bureau's motor cars and Diesel trucks and perform entirely satisfactorily. Likewise, large-scale tests in Diesel locomotives have proved shale-oil Diesel fuel to be wholly suited for this use.

Shale Economics Are Good

By virtue of these technical advances in mining, retorting, and refining, oil shale now occupies a very interesting economic position. The Bureau estimates that, with a capitalization of 50 percent equity and 50 percent borrowed funds and with all products selling at market values, the rate of return on the equity capital would be 11.2 percent after interest charges and income taxes. This return is based on an industry-scale operation and includes the cost of a pipeline to the west coast. Although this return is less than the average return of the petroleum industry, as indicated by published figures, it is high enough to warrant serious attention. Furthermore, oil-shale operations would not involve the exploration risks incident to crude-oil production.

Shale Requires Less Steel Than Petroleum

An industry-scale oil-shale operation, including a pipeline to the west coast, as well as all plant facilities, would require about 3 tons of construction steel per barrel of product. This means that substantially less steel is required to produce fuels from oil shale than to establish new fuels production from petroleum, including exploration and drilling. The fact that oil shale could be utilized to expand liquid fuels production with actual savings in steel is particularly significant now, when the foreign situation is unstable and the defense program is placing a heavy drain on our steel supplies.

Construction of Prototype Plant Encouraged

Sooner or later this Nation will rely in part on synthetic liquid fuels, and from the standpoint of security it is essential that it be fully prepared when the time comes.

The Department of the Interior believes that it would be prudent for private industry to establish a pioneer commercial oil-shale plant. The vast oil-shale resources, lying as they do within our own borders, assure a steady flow of oil in almost any emergency. Such a plant would prove the potentialities of America's oil shale resources and would justify increasing present estimates of proved oil reserves by many billions of barrels.

New Gas-Synthesis Process Developed for Oil-from-Coal

The progress made in developing methods for converting coal into liquid fuels parallels that for oil shale.

The gas-synthesis process, by which gas from coal, consisting of hydrogen and carbon monoxide, is converted into liquid fuels, was used commercially by the Germans. However, their plants left much to be desired. Sweeping technical improvements were necessary before the process could be considered for use in this country, because the German reactors required large quantities of steel and were very expensive. Furthermore, the largest reactor they used had a capacity of only 20 barrels a day, which meant that a commercial plant of several thousand barrels capacity would require a multitude of reactors with attendant high requirements of manpower and materials.

Opinion in the Bureau held that successful development of an acceptable gas-synthesis process depended on finding an efficient, rapid method of removing the tremendous quantities of heat evolved in the reaction. If this problem could be solved, permissible throughputs would be increased greatly; and as a result, material and manpower requirements would be reduced.

Approaching the problem with this in mind, the Bureau began experimenting on a small scale in its research laboratories at Bruceton, Pa. Rather than attempt to remove the heat from the reaction indirectly, as the Germans had done, the Bureau decided to dissipate the high heat of reaction directly by circulating cooling oil through the catalyst bed. Numerous problems were encountered in the initial experiments, and early results were discouraging. When experiments in the small, bench-scale equipment proved that the heat could be removed by this method, larger pilot plants of a 3-gallon-a-day capacity were built. Finally, successful development of a new process depended on the solution of one major problem. After the converter had been operated briefly, the bed of catalyst particles became cemented together, the circulating oil could not remove the heat generated, and, as a result, throughputs fell off sharply.

This difficulty was overcome by speeding the flow of cooling oil enough to keep the individual particles in constant motion, thereby preventing the bed from cementing together.

Upon gaining the necessary experience on the smaller pilot plants, the Bureau next constructed a larger 1-barrel-a-day pilot plant at Bruceton. This plant operated very satisfactorily and substantiated the previous development work.

Design and construction of a 50-barrel-a-day demonstration plant at Louisiana, Mo., were undertaken as rapidly as possible, and the plant was completed during the summer of 1951. A shake-down run was started on September 12; and, after necessary minor revisions were made, a comprehensive test run was started on October 23. This run, lasting 25 days, was entirely satisfactory. Some 75 to 85 percent of the synthesis gas was converted to liquid products in a single pass at a throughput six times greater than that attained in German reactors, and substantial quantities of gasoline and Diesel fuel were made.

High-Pressure Coal Gasification Advancing

Progress has been made toward developing an economical process for gasifying coal to make synthesis gas for the Fischer-Tropsch conversion and for generating hydrogen for direct hydrogenation of coal.

The latest and most important development is the gasification of powdered coal under pressure. A 500-pound-per-hour pulverized-coal gasifier using steam and oxygen and designed to operate at a pressure of 450 pounds has been constructed. Preliminary runs at 100 pounds pressure have been successful, and operating pressures are being increased gradually.

In addition to being the basic process for coal-to-oil operations, gasification also is the key to economical production of fertilizers from coal. The development of an inexpensive coal-gasification process very probably would open the door for large-scale fertilizer operations using coal as the raw material.

Commercial Gas-Synthesis Plants

After analyzing the progress that has been made, it is evident that the information necessary for designing commercial gas-synthesis plants starting with coal will be available soon. Demonstration of a high-pressure coal gasification process is the most important problem remaining. A low-cost gasification process would make an important contribution toward economical production of liquid fuels from coal by the gas-synthesis process.

American Equipment Used Successfully in Coal Hydrogenation

Coal hydrogenation, which involves direct reaction of coal with hydrogen to produce liquid fuels, already was a proved process when the Bureau's synthetic liquid fuels program was started, as the Germans had used it to produce virtually all of their aviation gasoline for World War II. However, coal hydrogenation was not in use in the United States; and, because the process operates at higher pressures than ordinary fuels plants, much of the specialized equipment had never been made in this country.

Probably the greatest achievement in coal hydrogenation has been the design and fabrication of new, and in many instances, improved equipment by American industry, and the use of this equipment in building and operating the Bureau of Mines Demonstration Plant at Louisiana, Mo. Although it would have been much easier to obtain the highly specialized equipment from Germany, it was decided to build a completely American plant. This required the cooperation of numerous manufacturers. As a result, industrial concerns are now in a position to supply such equipment. Improvements over German plants, especially in instrumentation and control, have been incorporated in the 200-to-300-barrel-per-day demonstration plant at Louisiana. Four American coals have been successfully processed in the plant, and the gasoline produced has been extensively tested by the Armed Forces and found to be very satisfactory.

Coal-Hydrogenation Cost Estimates

Bureau of Mines estimates for coal-hydrogenation production costs for two assumed locations, Rock Springs, Wyo., and western Kentucky, were completed during the year. The Bureau contracted with Ebasco Services, Inc., a prominent engineering, management, and consulting firm, to make an independent review of some of the more important costs included in these estimates.

Ebasco's evaluation included a detailed study of two important items of construction costs; an extensive analysis of six major operating cost factors; a comprehensive study of the marketability of chemical coproducts; and an investigation of company-financed housing requirements. Ebasco also studied methods of financing initial plants.

The Ebasco studies supported many of the important cost factors previously estimated by the Bureau. Following is a comparison of some of the factors estimated by Ebasco and the Bureau for an assumed plant at Rock Springs, Wyo., based on January 1951 conditions:

Comparison of cost estimates for a coal-hydrogenation plant at Rock Springs, Wyo.

(All figures are based on conditions as of January 1951)

	Bureau	Ebasco
Plant-capacity basis - barrels per calendar day	30,000	30,000
Power plant	$55,500,000	$51,400,000
Hydrogen compression and purification facilities ...	57,948,000	56,184,000
Company-financed housing and community facilities ..	5,000,000	250,000
Percent equity capital	40	50

Other important comparisons have been developed by adjusting the Bureau's figures to take account of the Ebasco findings as follows:

	Bureau original	Bureau figures revised to include Ebasco findings
Total capital investment	$414,440,000	$403,827,000
Annual production costs including depreciation	53,199,500	58,753,500
Annual value of fuels and chemicals, including gasoline at 11 cents per gallon	79,859,000	76,180,700
Average return on equity capital with gasoline at 11 cents per gallon	6.98 percent	3.59 percent
Production costs (before net income, interest charges and income taxes), per gallon of total product	1/ 10.2 cents	1/ 11.4 cents

1/ After credits of $6,254,000 income from sale of ammonium sulfate and sulfur.

It should be emphasized that the Ebasco evaluations represent its best judgment, based on independent studies. Ebasco obtained information from a wide variety of sources, including discussions on housing with individuals at the sites; information on equipment costs from industry; information from railroads and other agencies in product handling; information from the chemical industry and defense agencies on chemical demands; and information on manpower requirements from specialized industries, such as synthetic ammonia, petroleum refining, and others.

As indicated in the foregoing table, Ebasco's independent estimates for the power-plant and hydrogen compression and purification facilities, which account for about 25 percent of the total investment, were lower than the Bureau previously estimated. In its report Ebasco stated that these estimates, "constitute a close check of the Bureau's figures for these facilities and in effect confirm the original estimates and the accuracy of adjustments made by the Bureau."

In the matter of company-financed housing, previous Bureau studies had indicated that no provision for community facilities or employee housing would have to be included in the capital requirements of a coal-hydrogenation plant at Rock Springs, Wyo. However, the Bureau estimates did include an item of

$5,000,000 to assist the surrounding communities in providing the necessary facilities. After careful examination of all the factors involved, Ebasco concluded that existing communities near the plant site would be willing to provide the necessary facilities and that only $250,000 need be included in the capital requirements to assist local planning groups and to protect the interests of the plant and its employees.

To insure an accurate appraisal of operating costs, the Bureau requested Ebasco to draw up complete manpower tables, including maintenance and operating labor as well as administrative, supervisory and clerical help, and to base related operating costs on these manning tables. Ebasco's findings on these items raised the Bureau's estimates of over-all operating costs by 10 percent.

It is of interest to compare actual production costs, before net income, interest charges, and income taxes, per gallon of total product because the nontechnical factors of financial arrangements and income taxes on which there is a greater variation of opinion are not involved. On this basis, the Ebasco findings increased the Bureau's previous estimate of 10.2 cents per gallon of total product to 11.4 cents, after credits for the sale of ammonium sulfate and sulfur.

Based on chemical market surveys, the Bureau, in its previous estimates, took advantage of the versatility of the coal-hydrogenation process to produce aromatic hydrocarbons and other chemicals as well as fuels. Production of these materials improves the economics of the operation because they can be readily sold at prices much higher than motor fuel. The Bureau also included facilities for producing ammonium sulfate and sulfur from the waste streams.

Ebasco made a thorough study of the chemical market potentials over the next several years and, with only one exception - namely, m-p-cresol - concurred with the Bureau's estimates of the marketability of chemicals from a single plant.

It is apparent, from the factors discussed, that the independent evaluation of Ebasco lends considerable support to the correctness of the Bureau's coal-hydrogenation cost estimates.

It must be emphasized that all of the estimates compared are for a so-called conventional plant the design of which is more or less frozen as of January 1951 and does not include important improvements that now are considered feasible as a result of the Bureau's technical studies on coal hydrogenation. These improvements, combined with additional facilities for producing greater quantities of valuable chemicals, would make the first commercial plants more attractive investments.

The Bureau of Mines is now proceeding with detailed studies of a modernized plant, including recent technical improvements and facilities for increased production of chemicals to determine coal-hydrogenation economics under these more favorable conditions.

Steel Requirements for Coal Hydro Not Excessive

There is a widespread impression that steel requirements for coal hydrogenation are excessively high. Actually, on an equivalent product basis, coal hydrogenation requires about the same amount of steel per daily barrel of product as the petroleum industry, including exploration and drilling. Coal hydrogenation yields higher-grade products than those normally obtained from petroleum. For example, hydrogenation yields no residual fuel oil, but rather motor fuel, LPG, and substantial quantities of valuable chemicals such as benzene, toluene, xylene, phenol, and others. Considering the type of products, steel requirements for coal hydrogenation are reasonable.

Commercial Coal-Hydrogenation Plants Needed

Some of the basic considerations of security that apply to oil shale are also applicable to coal hydrogenation. There is little question but that some time in the future domestic crude oil will have to be supplemented with liquid fuels from both coal and oil shale. The job will not be done with either alone.

United States coal resources are plentiful and widely distributed over many sections of the country. Coal-hydrogenation plants could be located in several large consuming areas. Taking advantage of the flexibility of coal hydrogenation by producing maximum quantities of chemicals would improve the economics of the process greatly. The immediate establishment of one or two coal-hydrogenation plants by private industry, as advocated by the Department of the Interior, could make a major contribution of needed chemicals and would help lay the groundwork for large-scale production of liquid fuels from coal should this become necessary for national security. It appears, however, that something in the way of Government incentives to private industry may be required at this time. Ebasco in its findings made the following statement: "We do not believe it would be feasible to finance the projects discussed in the Bureau of Mines report dated October 25, 1951, with private capital under conditions prevailing at January 1, 1951."

A summary follows of 1951 operations at each of the synthetic liquid fuels laboratories and demonstration plants of the Bureau of Mines:

Summary of 1951 Operations

Oil from Coal

Demonstration Plants, Louisiana, Mo.

During the past year, construction was completed and operations were started in the second of two Coal-to-Oil Demonstration Plants on the banks of the Mississippi near Louisiana, Mo. Prototypes of a new industry, both plants convert coal or lignite to high-quality synthetic liquid fuels and chemicals. However, they employ fundamentally different processing methods - coal hydrogenation and gas synthesis - in serving as proving grounds for American coals, equipment, and process modifications.

The Coal-Hydrogenation Demonstration Plant, a two-stage unit that operates at high temperatures and pressures, completed its second full year of experimental production. Two extended liquid-phase operations and one vapor-phase operation were carried out last year, and a third liquid-phase run was started for a total operating time of 150 "stream" days to December 1.

First, approximately 2,500 tons of western Kentucky coal was converted in two steps to nearly 350,000 gallons of gasoline, which was shipped to the military services for testing. Next, 2,500 tons of Illinois No. 6 coal was processed into vapor-phase charging stock. Then, liquid-phase processing of 4,400 tons of western Wyoming subbituminous coal was begun late in October. The products of these latter two liquid-phase operations will be converted to gasoline during the next vapor-phase run.

Each run was successful and achieved its objectives. Products were evaluated in Bureau and commercial laboratories and also passed road tests without difficulty, both in Bureau-operated vehicles and military fleet operations. The good quality of the gasoline, together with the quantity of aromatics and tar acids in the products, was significant.

Two integrated runs were made in the Gas-Synthesis Demonstration Plant. The first, started on September 4 and terminated on September 12 after achieving all objectives, was an orientation run to determine the plant's operability, complete the training of operators, observe the functioning of equipment and instrumentation, and reach as high a conversion rate as possible. The second, started on October 23 and completed November 18, showed that the Bureau's process is feasible, the equipment workable, the catalyst apparently long-lived, and the conversions in accord with those anticipated on the basis of pilot-plant results at Bruceton, Pa. The gasoline and Diesel oil produced during the run now are being evaluated.

Coal-gasification studies were continued at Louisiana, Mo., on a demonstration scale, and test runs in the oxygen-blown coal gasifier developed by the Bureau at Morgantown, W. Va., produced promising results. The synthesis-gas purification unit also was tested and proved capable of continuously reducing the impurities in the feed gas to less than 2 percent carbon dioxide and well under 0.05 grain total sulfur for each 100 cubic feet of gas.

In another operation, 75,800 pounds of iron oxide synthesis catalyst was produced at a cost of 16 cents a pound, not including plant amortization. Tests in bench-scale, pilot-plant, and demonstration-plant operations have shown this material to be as satisfactory as any catalyst previously tested.

Three major engineering cost estimates were made during the year. The original coal-hydrogenation plant cost estimate, based on a daily capacity of 30,000 barrels, was revised to comply with higher 1951 costs and with the advances in technical knowledge achieved in 3 years of demonstration-plant work. Two other cost estimates were made for 15,000-barrel-per-day plants, one using a southern Colorado bituminous coal and the other a northern Wyoming subbituminous coal.

Market surveys were continued to establish price and capacity trends for aromatic hydrocarbons, tar acids, and tar bases.

Laboratories and Pilot Plants, Bruceton and Pittsburgh, Pa.

Research at the Bruceton, Pa., laboratories and pilot plants on the synthesis of liquid fuel from gasified coal (Fischer-Tropsch and related processes), on the production of liquid fuel by hydrogenation of coal (Bergius and related processes), and on the gasification of coal in a vortex reactor has led to developments of theoretical as well as practical importance.

Oil was synthesized from hydrogen and carbon monoxide in a barrel-per-day pilot plant, using an internally cooled moving-bed reactor and a fused-iron catalyst. Although this plant was 13 times as large as those previously employed, no unusual difficulties were experienced during its operation.

The same catalyst was used in a number of slurry tests in which gas is bubbled through a catalyst powder suspended in oil. A problem of the catalyst settling was solved by adding modified bentonite to the slurry, increasing its viscosity. Uninterrupted experiments lasting 2 and 3 months thus were made possible.

Experiments on the Fischer-Tropsch synthesis at 500 to 1,500 p.s.i.g. pressure, with a nitrided fused-iron catalyst, showed little change of product distribution as compared to 100 to 300 p.s.i.g. pressure. The rate of the reaction, however, was not as sensitive to pressure changes in the higher range as it had been in the lower range of pressures.

Addition of alkali to iron Fischer-Tropsch catalysts was found to decrease the rates of carbiding and rereduction and to increase the rate of carbon deposition after carbiding was essentially completed. A method of preparing fairly pure Hagg iron carbide has been developed.

A combination of various physical and chemical analytical techniques was used to determine the constituents of the Fischer-Tropsch synthesis products. A scheme of mathematical analysis was devised to correlate the product compositions obtained with various catalysts and over a wide range of operating conditions. This correlation also predicts the maximum amount of any fraction of the product - Diesel oil, for example - that can be obtained under the most favorable conditions.

A pilot plant has been built for a continuous process of hydrogenating coal in a fluidized bed. Several coals also were tested in small autoclaves to determine their suitability for hydrogenation in the demonstration plant at Louisiana, Mo. Some were acceptable as received, while others first had to be dried and cleaned.

A study of the operating variables of the coal-hydrogenation process has shown that nickel, as nickel chloride, is the most suitable of the catalysts tested. It may be used in low concentration and produces a large amount of oil. A method for quantitative evaluation of the quality of coal-hydrogenation catalysts was developed.

For better utilization of the less-desirable products of coal hydrogenation, studies also are under way to find the best conditions (1) for obtaining gasoline by hydrogenation of the middle oil and (2) for recovering oil from the heavy-oil purge stream by steam stripping or coking. Preliminary results are promising in both instances.

New analytical procedures were devised for determining the composition of the products from coal hydrogenation, particularly that of the gasoline, tar acids, and tar bases.

Many of the variables of coal gasification, using steam and oxygen in a vortex reactor, have been explored and correlated. Introduction of part of the oxygen with the coal resulted in increased production of gas under otherwise equal conditions. The gasification reaction was diffusion-controlled above about 2,200° F., and complete gasification of coal appears possible in an adiabatic reactor under such conditions. With an optimum ratio of 7 to 7.5 cubic feet of oxygen per pound of coal, it is estimated that 34.1 pounds of coal and 247 cubic feet of oxygen at standard temperature and pressure are needed to produce 1,000 cubic feet of synthesis gas, a carbon monoxide-hydrogen mixture.

Abstracts of articles and patents pertaining to synthetic liquid fuels were compiled and published until recently, when lack of funds compelled suspension of this activity. A book on the Fischer-Tropsch reaction was published, and a bibliography on this process is being completed. The first two parts of a similar bibliography on coal hydrogenation were issued and the last part is being readied for printing. Reviews on the status of various aspects of this program were contributed to several journals, and translations of foreign articles of unusual interest in this field were made available as Bureau of Mines publications.

Synthesis-Gas Laboratory, Morgantown, W. Va., and Field Tests, Gorgas, Ala.

Planning for commercial-scale plants requiring large quantities of synthesis gas, both in this country and abroad, has emphasized the importance of the new developments in coal gasification. At Morgantown, W. Va., intensive work has been conducted on experimental development of pulverized-coal gasification with oxygen and steam. A high-pressure powdered-coal gasification process now being developed promises substantial reductions in the cost of synthesis gas, a major item of expense in manufacturing synthetic liquid fuels, ammonia, alcohol, and other chemical intermediates.

Experimental powdered-coal gasification units at the Morgantown station include:

(1) A small-scale gasifier handling about 50 pounds of powdered coal an hour and consisting essentially of a vertical tube about 6 feet high and 6 inches in internal diameter. This gasifier has provided data on the conditions under which coals can be gasified efficiently and also has proved valuable for evaluating the suitability of various types of coal for gasification.

(2) A new atmospheric-pressure gasifier, handling about 500 pounds of coal an hour, was completed during the year. Its design was worked out in

cooperation with Babcock & Wilcox Co., which also fabricated the unit. The performance of this gasifier with various arrangements of burners is being studied.

(3) A pressure gasifier completed in 1951 promises substantial economies in synthesis-gas production through reducing gas-compression costs. Designed to gasify 500 pounds of coal an hour at an operating pressure ranging up to 30 atmospheres or approximately 450 p.s.i., this unit in preliminary runs at 7 atmospheres showed high capacity, but rather severe erosion of the refractory lining occurred.

(4) Initial test runs in a pulsating flow or vibratory gasifier achieved extremely high throughput rates on the order of 1,000 pounds of coal an hour for each cubic foot of gasifier volume. However, erosion of the refractory lining again was experienced.

Gas-purification work at Morgantown included bench-scale experiments at elevated pressures to study the performance of (1) a copper-chromium-vanadium catalyst for simultaneous removal of hydrogen sulfide and organic sulfur from synthesis gas and (2) a copper catalyst for removal of small amounts of oxygen from synthesis gas.

As the recovery of sulfur must be a feature of the gas-purification system in any large Fischer-Tropsch plant, purification pilot-plant runs were made at 300 p.s.i.g. to study the selective removal of hydrogen sulfide from gases containing relatively large quantities of carbon dioxide. Among the purifying absorbents studied were triethanolamine, sodium and potassium carbonate, and tripotassium phosphate. Carbon dioxide is believed to have a deleterious effect on the Fischer-Tropsch synthesis, and part of the purification program dealt with the removal of this compound from synthesis gas by using diethanolamine. Throughout most of the operations in the purification pilot plant, the total sulfur content of raw synthesis gas was reduced to less than 0.05 of a grain per 100 cubic feet.

At Gorgas, Ala., the second underground gasification experiment was completed. Continuous operation had been maintained for 22 months, and the coal underlying 2 acres of land adjacent to the original 1,450-foot entry was consumed. Drilling new inlets or outlets tangent to the perimeter of the burned-out area made it possible to extend the gasification over areas of coal land and also to improve the quality of the products. The experiment showed the importance of attaining good contact between the air or oxygen and the reacting coal faces. At the conclusion of the second experiment, the underground system was flooded with water and the fire extinguished.

A third experiment was started at Gorgas in which all underground mining was eliminated by using an electrolinking process tried earlier by the Sinclair Coal Co. and the Missouri School of Mines. Electrodes were inserted in the coal bed through boreholes drilled both for their installation and for subsequent use as air or gas inlets and outlets. A potential was applied to a pair of electrodes, causing a current to flow. The electric current heated and carbonized the coal, and the formation of coke increased the permeability of the coal bed. When the resistance of the system was reduced sufficiently, the electric current was shut off and air was pumped underground to carry on gasification. An initial electrical linkage was formed between boreholes 67 feet apart and subsequently between boreholes 200 feet apart.

Gasification of a 67-foot section opened by electrolinking has been continued for 4 months with the production of combustible gases. As the throughput has been small, additional electrolinking will be attempted in the immediate future to increase the throughput and develop the system further.

Oil from Oil Shale

Experimental Mine, Rifle, Colo.

As problems associated with mining oil shale at low cost are nearer solution than other phases of the program, the scope of work was reduced considerably in 1951 at the Experimental Oil-Shale Mine on Naval Shale Reserve No. 1 near Rifle, western Colorado. Research activities were curtailed, and operations were directed primarily toward supplying the processing plant with oil shale.

Collection of drill cuttings from oil wells penetrating the Green River oil shale formations in the Rocky Mountain area was continued, but no additional data on potential reserves are available yet. As reported in 1950, an area of 1,000 square miles in Colorado could yield 300 billion barrels of shale oil from an oil-shale measure averaging 15 gallons per ton. Within this measure is the richer 30-gallon-per-ton Mahogany ledge that could yield nearly 100 billion barrels of shale oil.

The two sets of workings at the Experimental Oil-Shale Mine are known as the Selective Mine and the Underground Quarry. Initial production was from the Selective Mine, but the Underground Quarry has been supplying shale for the processing plant since 1949. Approximately 50,000 tons was mined from the Underground Quarry in 1951, and some 19,000 tons of this was transported to the processing plant.

In the mining and research program emphasis has been shifted to fundamental research. To this end, data were collected on the action of explosives by means of instrumentated tests and motion-picutre studies.

The drilling phase of the mining-research program involves two separate investigations, percussion and rotary drilling. Several facts were recorded on percussion-drilling fundamentals, and the study of heat-treating procedures for minimizing drill-rod failures was continued, with notable success.

Rotary-drilling research showed that the bench level of a commercial oil-shale mine could be drilled by rotary methods at a considerable saving over percussion methods. Estimates are that bit and drill rod costs for rotary methods would be about $0.0_ per foot, compared to about $0.028 per foot by percussion methods. In addition to the direct saving in bits and drill rods, the higher drilling rates achieved by rotary methods would lead to savings in such items as labor and power.

An exhaustive and continuous study of roofstone behavior has been conducted in an experimental test room in the Selective Mine since early 1947, when specialized equipment and apparatus were installed. By November 1948 the test room was 80 feet wide by 100 feet long. An extension to this room was completed in June 1951, making the total length 200 feet. Two rock falls occurred in the original section of the enlarged test room in August 1951, each consisting of a uniform slab 18 to 22 inches thick. Stratascope examinations of the roofstone in the Underground Quarry showed that no partings had developed at the 18- to 22-inch level.

In November 1950, a cost estimate was prepared for mining, crushing, and conveying 19,200 tons of oil shale daily to a processing plant. During 1951, revisions of this proposal were made to raise cost items to the January 1, 1951, price level. The revised estimated total cost of mining, crushing, and conveying oil shale to the retort stockpile is 47.63 cents per ton. This estimate includes management, depreciation, and taxes, but does not include depletion, profit, nor expenditures for off-site facilities.

Demonstration Plant, Rifle, Colo.

Pilot-plant activities in 1951 were concentrated on the gas-combustion process, a development of the Bureau's Oil-Shale Demonstration Plant near Rifle, Colo. In this continuous method of retorting oil shale, combustion of low-B.t.u. gas generated in the retort provides most of the necessary heat. All heating and cooling take place between gas and shale within the retort. Thus, neither exterior heat-exchange equipment nor cooling water, a scarce commodity in the West, is required. Furthermore, good yields are obtained at high retorting rates.

To evaluate the performance of the 6-ton-a-day gas-combustion pilot retort, a 4-day run was made on shale averaging 30 gallons a ton by Fischer assay, or about the grade that would be charged in a commercial operation. During the evaluation run, a yield of 95.7 percent by volume of Fischer assay was achieved at a retorting rate of 229 pounds an hour per square foot of cross-sectional area in the retort.

Related pilot studies included developing means of controlling oil-mist formation in the product cooling zone of the retort, investigating gas-air mixers, and testing equipment for separating the oil-mist from the gas stream.

Plans for a demonstration-scale gas-combustion retorting plant with a nominal capacity of about 300 tons a day were undertaken as soon as pilot-plant investigations had demonstrated that the process was sound technically. Through competitive bidding, the Blaw-Knox Construction Co., Chemical Plants Division, was awarded a contract for the design, fabrication, and erection of the demonstration unit, and it is anticipated that the new plant will be ready for operation in the summer of 1952.

Demonstration refining operations in 1951 provided much of the information needed for successfully adapting conventional thermal refining techniques to the processing of shale oil. Earlier runs in the experimental refinery had demonstrated the suitability of three thermal processes - visbreaking, recycle cracking, and coking - for making a virtually full range of liquid fuels from the crude material. Logically, the next step was to ascertain, for those processes, the exact procedures and operating conditions that would give the best product quality and yields. Test operations also were conducted on thermal reforming, a process not investigated previously.

In an operation simulating the production of liquid fuels from oil shale on an industrial scale, a crude oil suitable for pipeline transportation was produced by visbreaking. Recycle cracking is an attractive method of refining the visbroken crude, because this process gives high yields of gasoline and permits the use of available thermal equipment at existing refineries. In a recycle cracking run on crude shale oil, a naphtha yield of 49.6 volume percent of the charge was attained, together with 45.9 volume percent residuum.

Experimental treating operations were continued in an effort to determine procedures and operating conditions by which the minimum amount of reagents consistent with desired results would be consumed. Recent addition of tetraethyllead blending facilities to the plant made possible the production of shale-oil gasoline that is being used in automobiles and buses at the station. Shale Diesel fuel has been in use on the project for almost 2 years. The performance of both appears to be equal to that of the corresponding petroleum products.

Through cooperative agreements, interested organizations contributed valuable additions to oil-shale technology. Sixteen new agreements were negotiated during the year, and four existing agreements were extended.

Under one new agreement, the Geneva Steel Co. tested a high-melting-point shale-oil pitch to determine its suitability as a blending component for preparing metallurgical coke from Utah coal. Under another, shale Diesel fuel was tested in a locomotive of the Denver & Rio Grande Western Railroad over a period of 751 hours. In both instances, results were encouraging.

Detailed cost estimates were made during the year for a nominal 250,000-barrel-per-day industry-scale oil-shale operation utilizing the newest developments in mining, retorting, and refining. The estimates include transportation costs to the West Coast and are based on the Bureau's oil-shale-mining demonstration work and the gas-combustion retort developed by the Bureau. Two refining plans were considered, one incorporating basic thermal cracking and the other using a mild hydrogenation process developed by Union Oil Co. of California. Both refining options yield specification products; and, as would be expected, the hydrogenation process produces high yields of premium gasoline and Diesel oil.

Based on a capitalization of 50 percent equity and 50 percent borrowed funds, and with all products selling at current prices, the rate of return on the equity capital is estimated to be 8.4 percent after income taxes for the plan incorporating thermal refining and 11.2 percent when using mild hydrogenation to refine the crude oil. Although the thermal refining option is less economical, it has the advantage of utilizing refining processes that are in use at the present time and therefore would permit processing viscosity-broken crude shale oil in existing refineries.

Steel requirements for the industry scale operation, including mining, retorting, refining, and a pipeline to the west coast, are 579,000 tons, or about 3.0 tons per daily barrel of liquid fuel for the thermal cracking option and 555,000 tons, or about 2.8 tons per daily barrel of liquid fuel for the mild hydrogenation option. The thermal-cracking plan requires a larger pipeline, hence the higher steel requirement.

Laboratories and Pilot Plants, Laramie, Wyo.

For two major reasons, the attention of the staff at the Bureau's Oil-Shale Experiment Station in Laramie, Wyo., was centered during the past year on development of a process for retorting oil shale at high temperatures: (1) Critical chemical products now in short supply can be produced in this manner, and (2) the type of retort that appears most suitable for this high-temperature operation also shows promise of extremely high throughputs in the normal retorting temperature range.

Before tentative design for a small pilot plant was developed, three methods of retorting at high temperatures were studied: (1) The inclined-surface retort, somewhat similar to a conventional coal-carbonization retort except that the retort tube was set at an angle to permit continuous flow of shale; (2) a single-vessel fluidized-bed retort; and (3) an entrained-solids retort in which the shale is carried into and out of a heated, vertical furnace by an entraining gas or steam. Of the three methods, the entrained-solids retort appears to offer the best possibility of enlargement to pilot-plant and commercial-plant size, and a small pilot plant employing this principle is now being constructed.

In refining research, efforts were directed toward developing methods for treating thermally cracked shale gasoline and improving distillate stocks for use either as Diesel fuels or catalytic cracking stocks. As background for the gasoline treating research, extensive studies were made of the properties of this type of gasoline and the polymer formed when it is acid treated. This work has resulted in a catalytic treating method that appears to have advantages over those previously used. It indicates also that low-temperature acid treating itself may be essentially a catalytic-treating process and suggests lines of investigation that may result in improved acid-treating techniques.

Solvents have been found that are more effective in preparing catalytic-cracking stocks and Diesel fuels from shale-oil distillates than those studied in the past. Equipment has been constructed for use in extending the study of hydrogenation of shale oil and shale-oil distillates to pressures above 1,500 p.s.i., the maximum safe operating pressure for equipment previously available. Now virtually ready for operation, this new equipment will permit more-complete evaluation of the hydrogenation process as a method for refining shale oil.

Compositions and properties were determined for two series of oil shales from the Experimental Mine that assayed from 10.5 to 75 gallons of oil a ton and were representative of minable beds in the Mahogany ledge. The data, published in a Bureau of Mines report of investigations, included the results of petrographic and X-ray examinations, determinations of shale properties, ultimate and oxide analyses, assays and examinations of assay products, and weathering tests.

The constitution of organic material in Colorado oil shale was studied by degrading it into smaller organic fragments for analysis and identification. The degradation methods included oxidation by alkaline potassium permanganate and nitric acid, hydrolysis with hydrochloric acid and potassium hydroxide, extraction with organic solvents, and destructive distillation, or heating. The results indicated that the organic material consisted preponderately of nonbenzenoid structures. Aromatic and terpene structures appeared to be present in minor amounts, but they were either of low molecular weight or were readily susceptible to oxidation.

Knowledge of the composition of shale oil still is sketchy, but it is vastly greater than that available a few years ago. It has been augmented during the past year by publications on pyrroles identified in shale-oil naphtha, gum formation in shale-oil naphtha, and determination of nitrogen in shale oil. Presentations before technical societies included papers on mass spectral correlations in composition studies, composition of crude shale oils, and oils from high-temperature retorting.

Recent analytical research has emphasized composition studies on three types of shale-oil products, the oils from high-temperature retorting, and the primary naphtha and heavy gas-oil fractions from N-T-U crude oil. In addition, work has been done on the development of analytical methods.

Tar acids recovered in refining thermally cracked gasoline from N-T-U crude shale oil were purified by removing the nitrogen- and sulfur-containing constituents, tar bases, carboxylic acids, water, and polymers to produce phenol, cresols, and xylenols of commercial quality.

Tar bases, also a byproduct of shale-oil gasoline refining, were treated to remove impurities and stabilized to reduce gum and color formation. Tests showed that the purified bases, when added to refined shale-oil gasoline, had no detrimental effect on its stability, and an increase in the octane rating of the gasoline was realized.