

by selective adsorption on silica gel. The pure types of hydrocarbons are then distilled in one of several vacuum-jacketed precision glass columns. For the identification of the pure fractions obtained by use of these columns, their characteristic physical constants (freezing point, refractive index, etc.) and the melting points of pure, crystalline derivatives are measured. Most aromatic polynuclear compounds such as are contained in coal hydrogenation oils form complexes with picric acid and with 1, 3, 5,-trinitrobenzene. These complexes usually are crystalline solids with sharp melting points. The use of picric acid and trinitrobenzene for the characterization of high molecular weight polynuclear compounds suffers from the disadvantage that the hydrocarbon is relatively only slightly soluble and often precipitates before the complex, or it is precipitated in excess on the complex compound. It was found that 2, 4, 7,-trinitrofluorenone is a much better reagent, and the molecular complexes of this compound with 26 pure polynuclear hydrocarbons were prepared and their melting points determined.<sup>87</sup>

For the characterization of coal-hydrogenation oils boiling above 300° C., precision vacuum distillation is desirable. Because no satisfactory standards have been established for distillation efficiencies of vacuum stills, a search is being made for a two-component mixture of oils which is suitable for such standardization work. Vapor pressure data were obtained for cyclohexylcyclopentane, n-dodecane, and different mixtures of these two compounds. Similar data are being obtained for tridecane and dicyclohexyl.<sup>88</sup>

The presence in coal hydrogenation and coal carbonization liquid products of polynuclear hydrocarbons with several fused rings sometimes has been used as evidence that such structures exist in the original coal. The possibility that polynuclear hydrocarbons containing many rings may be formed by dehydrogenation and cyclization of polynuclear hydrocarbons containing a smaller number of rings, or of the alkyl derivatives of such smaller ring systems, has been investigated, and several such syntheses have been demonstrated.<sup>89</sup>

#### Characterization of Light Oil

Preliminary characterization of the light oil obtained as a minor product in the low-pressure hydrogenation of Bruceton coal was accomplished by

- <sup>87</sup> Orchin, M., and Woolfolk, E. O., Molecular Complexes with 2, 4, 7,-Trinitrofluorenone: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 1727-1729.
- <sup>88</sup> Orchin, M., and Feldman, J., The Isomerization of Dicyclohexyl: Jour. Amer. Chem. Soc., vol. 68, 1946, pp. 2737-2738.
- <sup>89</sup> Orchin, M., Apparatus for Small-Scale Vapor-Phase Treatment of Solid Compounds: Ind. and Eng. Chem., anal. ed., vol. 17, 1945, p. 673.
- Orchin, M., Aromatic Cyclodehydrogenation III. Experiments with 2-Ethylbiphenyl: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 571-2.
- Orchin, M., and Friedel, R. A., Aromatic Cyclodehydrogenation IV. Experiments in the Dinaphthyl Series: Jour., Amer. Chem. Soc., vol. 68, 1946, pp. 573-4.
- Orchin, M., and Reggel, L., Aromatic Cyclodehydrogenation V. A Synthesis of Fluoranthene: Jour., Amer. Chem. Soc. (In press.)

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separating the oil into neutral, acidic, and basic fractions.<sup>90/</sup> The tar-acid fraction was composed of about the same ratio of individual tar acids as the similar fraction obtained by high-temperature carbonization. The tar-base fraction contained large quantities of aniline and the toluidines.

Only the neutral oil boiling below 175° was investigated. Low-pressure hydrogenation of this fraction facilitated the separation of the neutral fraction into aromatic, paraffin, and naphthene-type compounds. After separation by selective adsorption on activated silica gel, the aromatic portion was found to contain toluene, ethylbenzene, xylenes, and a small quantity of benzene.

The paraffin-naphthene portion of the neutral fraction was estimated to contain 74 percent naphthenes and 26 percent paraffins. All the normal paraffin hydrocarbons from hexaneto decane were present. The naphthenes included cyclopentane and cyclohexane. Methylcyclohexane was the dominant component, being present to the extent of about 12.5 percent of the total paraffin-naphthene fraction. More than 40 percent of the total light oil boiled above 175° C.

#### FOREIGN FUELS

On October 1, 1945, the technologic functions and activities of the Liquid Fuels and Lubricants and the Solid Fuels Subcommittees of the Technical Industrial Intelligence Committee were given to the Bureau of Mines.

These subcommittees were established for the purpose of investigating the German technologic developments in their respective fields. The Liquid Fuels and Lubricants Subcommittee was organized in the fall of 1944, and its first group of investigators, under the direction of Dr. W. C. Schroeder, entered German synthetic oil plants as soon as they fell into the possession of the Allied troops early in 1945. Virtually the entire contingent of investigators arrived in Europe and began conducting full-scale investigations in German and liberated territory prior to V-E day.

The Solid Fuels Subcommittee was patterned along the same lines, and its first investigators, operating under the guidance of Dr. Harry F. Yancey, entered German mines and coal-processing plants prior to the cessation of actual combat.

The members of both subcommittees were recruited from the Bureau of Mines, the Petroleum Administration for War, the universities, and private industry.

The Liquid Fuels and Lubricants group, which consisted of the following 30 authorities in the petroleum field, conducted investigations on the many phases of oil production and refining. These included all production

<sup>90/</sup> Kaplan, E. H., Storch, H. H., and Orchin, M., Hydrogenation and Liquefaction of Coal. Part V. - Characterization of Light Oil: Bureau of Mines Tech. Paper 690, 1946, 18 pp.

and research work on aviation gasoline, motor gasoline, Diesel and fuel oil, as well as such byproducts as synthetic lubricating oils, soaps, fats, detergents, alcohols, benzene, butadiene, and many others produced from oil, coal, and shale.

Liquid Fuels and Lubricants

Allen, John G.	Phillips Petroleum Co., Bartlesville, Okla.	Kuhne,
Atwell, Harold V.	The Texas Co., Beacon, N. Y.	Mackus
Baldeschwieler, E. L.	Standard Oil Development Co., Bayway, N. J.	Mandel
Bays, George S.	Humble Oil & Refining Co., Baytown, Tex.	Newmar
Carlsmith, Leonard E.	Standard Oil Co. of Louisiana, Baton Rouge, La.	Odell,
Cotton, Ernest	Gulf Oil Corp., Pittsburgh, Pa.	Powell
Doherty, Joseph D.	Bureau of Mines, Washington, D. C.	Schine
Evans, L. P.	Socony Vacuum Oil Co., Paulsboro, N. J.	Schro
Faragher, Warren F.	Houdry Process Co., Linwood, Pa.	Sherw
Fraser, Donald S.	Gulf Oil Corp., Pittsburgh, Pa.	von E
Haensel, Vladimir	Universal Oil Products Co., Riverside, Ill.	Voss,
Hirst, Lester L.	Bureau of Mines, Louisiana, Mo.	Weir,
Horne, William A.	Gulf Research & Development Co., Pittsburgh, Pa.	Roger
Jones, Irvin H.	The Koppers Co., Inc., Pittsburgh, Pa.	Peck,
Jones, Jean P.	Phillips Petroleum Co., Bartlesville, Okla.	Opal,

Kuhne, Paul K.	Gulf Oil Corp., Pittsburgh, Pa.
Mackusick, Byron L.	The Pure Oil Co., Winnetka, Ill.
Mandelbaum, M. R.	Kenyon & Kenyon, New York, N. Y.
Newman, Louis L.	Bureau of Mines, Washington, D. C.
Odell, William W.	Bureau of Mines, Washington, D. C.
Powell, Alfred R.	Koppers Co., Inc., Pittsburgh, Pa.
Schindler, Hans	Pure Oil Co., Winnetka, Ill.
Schroeder, Wilburn C.	Bureau of Mines, Washington, D. C.
Sherwood, Peter W.	Mellon Institute of Industrial Research, Pittsburgh, Pa.
von Elbe, Guenther	Bureau of Mines, Pittsburgh, Pa.
Voss, Ernst F.	Humble Oil & Refining Co., Houston, Tex.
Weir, Horace M.	Consulting Petroleum Engineer.
Rogers, Edward J.	Bureau of Mines, Pittsburgh, Pa.
Peck, E. B.	Standard Oil Development Co., New York, N. Y.
Opal, Earl	Petroleum Administration for War, Washington, D. C.

The solid fuels group, which consisted of eight men who are leaders in the field of solid-fuel technology, was engaged in conducting investigations on research and technology of mining, coal preparation, high- and low-temperature carbonization, byproduct recovery, complete gasification, including underground gasification; domestic and industrial coal and coke combustion, gas-turbine operation, and many other related subjects.

Solid Fuels

Buch, John W.	Bureau of Mines, Wilkes-Barre, Pa.
Driessen, Maximiliaan G.	Dutch State Mines.
Fraser, Thomas	Bureau of Mines, Washington, D. C.
Lowry, Homer H.	Carnegie Institute of Technology, Pittsburgh, Pa.
Reed, Frank H.	State of Illinois, State Geological Survey Division, Urbana, Ill.
Rose, Harold J.	Bituminous Coal Research, Inc., Pittsburgh, Pa.
Schmidt, Laurence D.	Bureau of Mines, Pittsburgh, Pa.
Yancey, Harry F.	Bureau of Mines, Seattle, Wash.

Although the major portion of the work of direct investigation of both teams was completed before the end of the fiscal year, it has been found necessary for two investigators to remain in Germany after the end of the year to continue with the investigation of a large volume of documents and the procuring of samples of catalysts and products and specimens of important pieces of machinery and equipment. Other investigators will be needed in the near future.

As a result of the investigations, many reports have been written, and documents supporting them have been accumulated. A few pieces of apparatus and many samples of catalysts and products have been collected. The information obtained covers the results of many years of research by leading technologists in Germany. To obtain this information independently, at least 5 years of research work by the staffs of many of the petroleum and coal research laboratories in the United States and the expenditure of many millions of dollars would be required.

The technical files and the samples of equipment, catalysts, and intermediate and end products were turned over by the Liquid Fuels and Lubricants and the Solid Fuels Subcommittees to the Bureau of Mines, which has the responsibility of indexing and digesting the files, studying the equipment and analyzing the catalysts and products, and releasing the information to the public in accordance with Presidential Orders 9568 and 9604, which direct that, so far as it may be done without prejudice to public interest, industrial information withheld from public dissemination for the purpose of national military security be released to United States industry.

To accomplish this end, the Foreign Synthetic Liquid Fuels Division was established to process and maintain information on both liquid and solid fuels. Facilities were provided for storing and examining microfilm reels of the source documents captured in Germany. Liaison was established with the Joint Intelligence Objectives Agency, the War and Navy Departments, and the Office of Technical Services (previously known as Office of the Publication Board) in the Department of Commerce to obtain copies of all reports relating to fuels and lubricants and other subject matter for distribution to the various branches of the Bureau of Mines and for reference collection open to the general public. Considerable use has been made of this library by employees of coal, oil, and chemical companies, particularly of their research organizations.

For the benefit of those who could not conveniently make use of the library facilities, arrangements were made for the Library of Congress to provide copies of the microfilm reels. By the end of the year, 153 reels covering the entire field of interest of both subcommittees were deposited in the Library of Congress, and indexes to these reels, together with instruction for their purchase, were sent out to a mailing list compiled by the former Technical Advisory Committee of the Petroleum Administration for War.

To make the reports written by the members of the investigating teams more readily available outside of the library of the division, the reference copies relating to fuels and lubricants were reproduced on microfilm reels, which have been added to the series on deposit in the Library of Congress. Many of these reports have been previously distributed in mimeographed form prepared by the Technical Advisory Committee. Three of them have been reproduced by the Bureau of Mines and distributed to the mailing list and to visitors to the library. 91/, 92/, 93/

The availability of these reports has been announced in the Bibliography of Scientific and Industrial Reports issued weekly by the Office of Technical Services in the Department of Commerce. Every opportunity was used to publicize this bibliography and the services available in the Office of Technical Services in correspondence relating to reports and at meetings of technical societies and trade associations such as the American Institute of Mining Engineers, American Chemical Society, American Petroleum Institute, and the American Byproduct Coke Institute.

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- 91/ Holroyd, R., Report on Investigations by Fuels and Lubricants Teams at the I. G. Farbenindustrie A. G. Works at Leuna: C.I.O.S. Report XXXII-107, Item 30, 1945, 140 pp.; Bureau of Mines Inf. Circ. 7370, 1946, 135 pp. T.O.M. Reel 197.
- 92/ Hollings, H., Wintershall A. G., Lützkendorf, near Muehlen, Germany: C.I.O.S. Report XXXII-90, Item 30, 1945, 37 pp.; Bureau of Mines Inf. Circ. 7369, 1946, 21 pp. T.O.M. Reel 197.
- 93/ Faragher, W. F., and Horne, W. A., Manufacture and Regeneration of Catalysts at I. G. Farbenindustrie Ludwigshafen/Oppau: F.I.A.T. Report No. 422, 1945, 6 pp.; Bureau of Mines Inf. Circ. 7368, 1946, 6 pp. T.O.M. Reel 199.



Emphasis must be placed on the fact that the reference library at its inception consisted of manuscript copies of the reports furnished by the investigators long before the machinery could be established for the reproduction of these items. Similarly, approximately 90 percent of the microfilm material was obtained prior to the establishment of the Field Intelligence Agency - Technical at Höchst (FIAT), from which a steady flow of reports and microfilms is being received.

With the active sponsorship of the American Petroleum Institute, a Film Study Group was established for the purpose of scanning the microfilm reels and abstracts, and, where warranted, to completely translate the material appearing on the microfilm reels. Reports on approximately 50 reels were completed by the Film Study Group before the end of the fiscal year. Thirty-one of these reports have been reproduced on microfilm reels, which are included among those on deposit in the Library of Congress.

Provisions for examining the samples of catalyst and intermediate and end products have been made by a special committee established by the Technical Advisory Committee of the Petroleum Administration for War. The active work of distributing the samples to the appropriate analytical groups has been conducted by the organization functioning in Pittsburgh under the supervision of Dr. H. H. Storch. Results of these analyses have just begun to come in and will also be disseminated by the microfilm route.

The activities of the division were well-received by the mining, oil, gas, and chemical industries, government agencies, and the universities. The value of this work is attested by the number of visitors making extensive use of the library facilities of the division and the prodigious amount of work donated by the Film Study Groups, which are composed of teams operating within the research organizations of some 30 leading oil companies, fuel-processing-equipment companies, and the Bureau of Mines. This is further attested by the enthusiastic cooperation of the groups in the analytical laboratories of the oil companies, the Aberdeen Proving Grounds of the U. S. Army, the Bureau of Standards, and the Bureau of Mines, who are voluntarily working on the samples of catalysts, oils, greases, and a large variety of chemicals.

#### Literature Translations and Abstracts

The Research and Development Division has been translating and abstracting information on synthetic liquid fuels contained in captured German documents. A preliminary report on the status of German development work on the Fischer-Tropsch and related processes was published.<sup>94/</sup> Translations were made of documents that present in some detail the results of research work

<sup>94/</sup> Storch, H. H., Powell, A. R., and Atwell, H. V., Fischer-Tropsch Report No. 1 of the U. S. Government Technical Oil Mission, Sn-MC-1, T.O.M. No. 5.

on the Fischer-Tropsch process done at the Kaiser-Wilhelm Institute für Kohlenforschung<sup>95/</sup> and by the I. G. Farbenindustrie, A. G.

#### Coal-Hydrogenation Information from Captured German Documents

The information from Germany has been rather complete in providing a description of the German Synthetic Fuel Industry. The picture has been clarified quite well as regards plant and equipment operation and production problems. In addition, a great deal of factual data on unit and equipment design with, in many instances, material and heat balances, have permitted calculation of approximate thermodynamic and physical constants, heat-transfer rates, and heats of reaction. The use of this calculated information is assisting in the evaluation of necessary design data for the demonstration plant.

The mechanical engineering information gleaned from the Germans has assisted us by giving us some information on high-pressure design, particularly on handling erosive fluids. Rather complete drawings of valves, piping details, converters, and preheaters often have had more than mere descriptive value. The use of rather low alloy-content steels for exceptionally severe service has been of great metallurgical interest.

Fortunately, there is enough information to make possible a rather accurate economic study of German operating costs. These are valuable to our Research and Development Program as a base case for determining the economic impact of changes and as an orienting influence on our attempts to improve the process. These data are being organized and studied very carefully in Pittsburgh.

- 95/ Leva, M., Translations of German Documents on the Development of Iron Catalysts for the Fischer-Tropsch Synthesis. Part I. T.O.M. Reel 101. Doc. P. G. 21559-NID, "Report on the Middle-Pressure Synthesis with Iron Catalysts, June 1940," 51 pp.; Doc. P. G. 21574-NID, "Lecture and Discussion on Iron Catalysts for Middle-Pressure Synthesis by Dr. H. Pichler, with a preface by Director Franz Fischer, Sept. 9, 1940," 21 pp.; Doc. P. G. 21581-NID, "Recent Investigations on Iron Catalysts," 98 pp.; Doc. P. G. 21577-NID, "Research on the Behavior of Iron Catalysts when Operated with H<sub>2</sub>-Rich Synthesis Gas," 8 pp.
- Leva, M., and Atwell, H. V., Part II. T.O.M. Reel 101. Doc. P. G. 21576-NID, "Process for the Production of Hydrocarbons (Patent Application)," 10 pp.; Doc. P. G. 21578-NID, "The Middle-Pressure Synthesis with Iron Catalysts, Sept. 9, 1939," 14 pp.; Doc. P. G. 21579-NID, "Investigation of the Reaction Water Obtained from Middle-Pressure Synthesis Experiments with Iron Catalysts and Hydrogen-Rich Starting Gas, F. Weinrotter, April 30, 1941," 12 pp.; T.O.M. Reel 134, Navy 5811, Item IB-23 (first half), "Hydrocarbon Synthesis with Iron Catalysts, Leuna Works, April 5, 1940," 12 pp.; T.O.M. Reel 86, Bag 3979, Item 78, "Experiences with the Semi-Commercial Synol Research Plant ME-458 Report No. 472/44A, Leuna Works, October 10, 1944," 22 pp. Copies available in "ditto" form at Pittsburgh, Pa.



Although many engineering details of design and operation of German coal-hydrogenation plants are contained in captured documents, there is little information on fundamental process research. Only one film (Technical Oil Mission Reel No. 145) contains a record of research in the I.G. Farben Hochdruck Laboratory. The subjects discussed in these documents concern the behavior of various coals and a multitude of "trouble-shooting" experiments designed to solve immediate production problems, but no systematic research designed to elucidate the fundamental chemistry of the process.