

## INTRODUCTION

From July 1, 1949, to July 1, 1950, the Bureau of Mines conducted the research and technologic work on coal and coal products that is summarized in this report. The research, described in this fifteenth of a series of résumés, is presented in more detail in publications (see footnote references) that appeared during the fiscal year, though in some cases research data are given that have not been published elsewhere. Results of research in the experimental coal mine, on explosives, and on gas and dust explosions will be found in Bureau of Mines Report of Investigations 4228, Annual Report of Research and Technologic Work on Explosives, Explosions, and Flames, Fiscal Year 1950.

An over-all view of our coal, oil, and natural-gas reserves was presented, and the future possibilities for the production of synthetic liquid fuels from coal were delineated.<sup>3/</sup>

A study of future petroleum supply and demand<sup>4/</sup> indicated that by 1975 the bulk of our oil may have to come either from foreign countries or from alternate domestic sources such as coal. Coal, the most abundant alternate source of liquid fuels, can be converted to gasoline and oils by two processes. Quantities and characteristics of coal requisite for each process were discussed, and cost data for products were presented. The current status of the Bureau of Mines demonstration plants for the production of synthetic liquid fuels from coal and new developments in processes for such production were reported.<sup>5/6/</sup>

Current and future aspects of chemistry in the coal industry were considered,<sup>7/</sup> with a brief résumé of the present situation in coal reserves and production.

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- 3/ Fielzner, A. C., The National Fuel Reserves and Future Fuel Supplies: Coal Mining Modernization Handbook, 1949, pp. 374-390.
  - 4/ Doherty, J. B., Synthetic Oil from Coal: Colorado School of Mines Quarterly, vol. 45, No. 2B, April 1950, pp. 77-95; discussion pp. 95-105.
  - 5/ Schroeder, W. C., Synthetic Liquid Fuel - Two Coal-to-Oil Demonstration Plants Are Producing Liquid Fuels from Coal on the Minimum Scale Enabling the Bureau of Mines to Make Available to Industry the Necessary Cost and Engineering Data for Commercial Operations: Armed Forces Chem. Jour., vol. III, No. 5, July 1949, pp. 12-13.
  - 6/ Schroeder, W. C., Synthetic Fuel Production: Petrol. Eng., vol. 22, May 1950, pp. A-53, 56, 60, 63, 64, 66, 68.
  - 7/ Hunter, T. W., Mineral Fuels (in Basic Minerals - Production High But Reserves Cause Concern): Ind. Eng. Chem., vol. 42, June 1950, pp. 978-983.

Progress in coal-mine safety research from 1910 to 1950 was reviewed.<sup>8/</sup>

Construction of the first wing of the Anthracite Research Laboratory at Schuylkill Haven, Pa., was completed, and research programs were initiated, while construction of the Lignite Research Laboratory at Grand Forks, N. Dak., was well under way.

#### SUMMARY

While total coal supplies were sufficient for the country's needs, certain areas lacked adequate supplies of solid fuels and their products for special purposes. The Bureau of Mines research program on the mining, preparation, and utilization of coal was prosecuted vigorously to insure development of reserves and supplies of such coals, without sacrifice of safety, efficiency, and conservation.

To clarify our reserve position and augment our dwindling reserves of coking coal, the Bureau continued its estimation of known minable reserves of such coals in central and southern Pennsylvania, southern West Virginia, and eastern Kentucky. This survey was supplemented by field investigations in the Georges Creek Basin in Maryland, where diamond drilling revealed estimated reserves of coking coal in excess of 399,000,000 tons, of which 50 to 60 percent is recoverable. In the laboratory, the problems of alleviating shortages of coal suitable for metallurgical coke were attacked by studies of the washing and blending characteristics of lower-grade coals, not now usable for coke making. These studies provided essential data on the carbonizing properties of 46 American coals and of blends of these coals to guide coke producers in selecting proper coals for their ovens. Research on the low-temperature carbonization of lower-rank coals was initiated, and a survey of the yields and properties of the products of such carbonization from representative western coals was made. Determination of the preparation characteristics of coals from Illinois, Tennessee, Pennsylvania, and West Virginia demonstrated the feasibility of upgrading many of these coals by preparation methods. Research on the desulfurizing of high-sulfur coals gave information on the possibilities of using ammonia gas for this purpose.

Conservation of our coal resources through efficient utilization was the goal of much of the Bureau's program. As in previous years, the Bureau of Mines acted as a consultant to other Federal agencies on the purchase of fuel, fuel-burning and boiler-room equipment, and on boiler-water chemical treatment. Analyses and recommendations were made on 9,335 samples of boiler water, and 580 Bureau of Mines field water-test kits, 10,259 bottles of chemical reagents, and 12,060 test-kit-replacement items were distributed to various Government activities. Proximate or ultimate analyses were made on 10,100 samples from purchases of coal and tippie and breaker inspections on which Federal fuel purchases were based. The savings resulting from these consulting services continued at a level many times their cost, and boiler-water research indicated even greater savings might be forthcoming in the future. Studies of the mechanism of external corrosion of furnace-wall tubes in boiler furnaces provided information that can greatly reduce the cost of equipment replacement and out-of-service time. In cooperation with the American Institute of Mechanical Engineers, the Bureau made further determinations of furnace-heat absorption in utility power boiler furnaces, which help to improve efficiency of operation, with consequent increases in fuel conservation.

<sup>8/</sup> Fieldner, Arno C., Achievements in Mine Safety Research and Problems Yet to be Solved: Bureau of Mines Inf. Circ. 7573, 1953, 31 pp.

Continued emphasis was placed on the development of feasible methods of utilizing our vast reserves of low-rank coals in the West, whose high natural-moisture content places them at a disadvantage in competition with higher-rank coals. Bureau investigations of drying these coals in the entrained and fluidized state promised to offset this disadvantage.

A new technique, involving the use of replica films, was developed for utilizing the electron microscope in studying the microstructure of coal. Shortages of montan wax for industrial use led to a survey of the waxes extractable from American lignites, with Arkansas and California lignites providing the highest yields of wax in solvent extraction tests.

Seeking to increase recovery and efficiency to meet primary problems of conservation in the coal industry, the Bureau studied the extraction of pillars underground with mechanized equipment and demonstrated that such extraction increases percentage recovery of coal, while decreasing production per mobile loading machine. In the anthracite fields of Pennsylvania, mechanization was advanced by successful underground tests on German shearing and packing machines and on the Bureau-developed scraper-shaker loader. Research was initiated on new methods of roof support in anthracite mines that will greatly reduce injuries to miners from roof falls. Another major problem in coal conservation was the huge quantities of coal consumed by uncontrolled mine fires. Sealing, flooding, and carbon dioxide were employed to control such fires.

Striving to achieve greater safety in mining coal, Bureau research yielded data on the effect of operating conditions on the composition of exhaust gases from Diesel engines used underground that is essential to the solution of many operating and combustion problems. To overcome harmful atmospheric conditions in coal mines, several studies were completed on the determination, collection, and dissemination of air-borne dusts and on methods of their control. More than 16,000 samples of mine air were analyzed, and over 1,000 samples of air-borne dusts were examined to determine the adequacy of ventilation in coal mines, as well as to detect and aid in the elimination of hazards from gases and mine fires. Eight new approvals and 23 extensions of approvals were granted for respiratory protective devices tested by the Bureau.

Research on the gasification of coal and lignite was continued, including the second underground gasification project at Gorges, Ala. In this experiment, the effect of various conditions of flow on the heating value of the gas produced has been determined. Experimentation with the atmospheric-pressure pilot plant to produce synthesis gas by the gasification of pulverized fuel reduced oxygen requirements for the gasification of coal to the lowest yet reported for any continuous process; a new unit was developed that permitted the testing of various types of coal for their utility in making synthesis gas on a commercial scale. An important phase of the gasification research was the purification of the synthesis gas for use in synthesizing liquid fuels. Laboratory and pilot-plant hydrogenation of coal to produce liquid fuels was studied in both liquid- and vapor-phase units. Greatly improved yields of soluble materials from coal resulted from using bicyclic hydroaromatic compounds, containing at least one phenolic hydroxyl group, as new solvent vehicles. Research to discover new, more readily available catalysts, as reactive as tin in coal hydrogenation, was particularly comprehensive. The factors involved in cleaning coal for hydrogenation to eliminate oil loss due to the presence of ash were studied.

At Louisiana, Mo., the Coal-Hydrogenation Demonstration Plant was completed and went through the necessary break-in procedures. The Gas-Synthesis Demonstration Plant was practically complete, and several of the units within the plant were successfully operated. As these processes demonstrated their technical feasibility, much interest was shown in the economic aspects of their application. Many estimates were made to determine the commercially competitive position of liquid fuels derived from coal.

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Washington, D. C.

##### Fuels and Explosives Division

A. C. Fieldner, chief  
L. C. McCabe, assistant chief  
Sidney Gottley, assistant to the chief  
R. L. Brown, coal technology coordinator, Coal Branch  
J. F. Barkley, chief, Fuels Utilization Branch  
W. H. Snyder, supervising engineer, Fuel Inspection Section, Fuels Utilization Branch  
W. C. Schroeder, chief, Office of Synthetic Liquid Fuels  
J. D. Doherty, assistant chief, Office of Synthetic Liquid Fuels

##### Health and Safety Division

J. J. Forbes, chief  
W. J. Pence, assistant chief  
G. H. Ash, chief, Safety Branch  
M. J. Ankony, chief, Coal-Mine Inspection Branch

##### Region I (Alaska)

S. H. Lorain, regional director, Juneau, Alaska

##### Region II (Washington, Oregon, Idaho, Montana)

S. M. Shelton, regional director  
H. F. Yancey, chief, Fuels Technology Division, Seattle, Wash.

##### Region IV (Wyoming, Utah, Colorado, Arizona, New Mexico)

J. E. East, Jr., regional director  
V. F. Perry, chief, Coal Branch, Golden, Colo.

##### Region V (North Dakota, South Dakota, Minnesota, Iowa, Nebraska)

Paul Zinner, regional director  
Alex C. Burr, chief, Fuels Technology Division, Grand Forks, N. Dak.

##### Region VII (Tennessee, North Carolina, South Carolina, Georgia, Alabama, Mississippi, Florida)

B. W. Gandrud, chief, Fuels Technology Division, Tuscaloosa, Ala.  
F. W. Smith, supervising engineer, Coke Section, Tuscaloosa, Ala.  
J. L. Elder, supervising engineer, Underground Gasification Project, Gorgas, Ala.

Region VIII (Maine, New Hampshire, Vermont, New York, Massachusetts, Connecticut, Rhode Island, New Jersey, Pennsylvania, Ohio, Indiana, Illinois, Maryland, Delaware, West Virginia, Virginia, Kentucky, and Louisiana, Mo.)

- H. P. Greenwald, regional director
- L. L. Hirst, chief, Coal-to-Oil Demonstration Plants, Louisiana, Mo.
- J. W. Buch, chief, Anthracite Subregion, Schuylkill Haven, Pa.
- H. M. Cooper, supervising chemist, Coal-Analysis Section, Pittsburgh, Pa.
- R. C. Corsy, supervising engineer, Combustion-Research Section, Pittsburgh, Pa.
- J. D. Davis, supervising chemist, Coal-Carbonization Section, Pittsburgh, Pa.
- W. A. Selvig, supervising chemist, Coal-Constitution and Miscellaneous Analysis Section, Pittsburgh, Pa.
- A. L. Toenges, supervising engineer, Bituminous-Coal Mining Section, Pittsburgh, Pa.
- A. A. Berk, supervising chemist, Boiler-Water Research Section, College Park, Md.
- L. Goldman, supervising chemist, Boiler-Water Service Section, College Park, Md.
- L. D. Schmidt, chief, Synthesis-Gas-Production Laboratories, Morgantown, W. Va.
- E. H. Storch, chief, Synthetic Liquid Fuels Research and Development Branch, Bruceton, Pa.
- M. A. Elliott, assistant chief, Synthetic Liquid Fuels Research and Development Branch, Pittsburgh, Pa.
- L. B. Berger, acting supervising chemist, Health Branch, Pittsburgh, Pa.

#### ORIGIN, COMPOSITION, AND PROPERTIES OF COAL

##### Inspection, Sampling, and Analysis

At the beginning of the fiscal year 1950, coal was being produced at a rate equal to or in excess of demand, and Federal agencies had no difficulty in obtaining bids to cover their requirements. Owing to the shift from a "seller's" to a "buyer's" market, all agencies were able to make their purchases on a guaranteed-analysis basis, including penalties for delivery of substandard coal. This method had been largely abandoned during the coal shortage of previous years. The Navy Department, purchasing coal for the Army, Air Force, and Navy, was the largest Federal coal-purchasing agency. In general, other departments purchase their own coal supply, except that the Federal Supply Service purchases the supply for some other agencies whose requirements are small. Contracting for coal for the fiscal year 1951 started in March and continued through the balance of the fiscal year.

Coal requirements of Federal agencies for the fiscal year were estimated to be approximately 5,000,000 tons. There was a large increase in requests from Federal agencies for analyses to be used in evaluating coal bids. This was caused in part by an increase in the number of bids received and in part by a more strict adherence to specification requirements that eliminated mines on which there were no Bureau of Mines analysis records.

At the urgent request of Federal agencies, mine operators, and coal-sales agencies for more service in obtaining mine-tipple analyses, the sampling program was expanded. A total of 1,505 tipple samples was collected at 481 mines in 12 States. At the beginning of the fiscal year, requests for sampling more than 600 mines were on file, with additional requests received daily. At the close of the year, about 350 mines still remained unsampled. For carbonization, grindability, electrode carbon, and other special tests, 447 samples were collected in 441 mines. At the same time, 36,000 tons of coal exported to Japan were inspected and sampled at Norfolk, Va.

To promote more accurate sampling of delivered coal and reduce sampling costs, many Government installations have installed mechanical equipment for preparing samples. It became necessary to issue new instructions<sup>9/</sup> for coal sampling to cover this innovation. At the request of the Navy Department and Marine Corps, special instructions were given in coal sampling at five installations.

Data in the first of the Bureau's series of technical papers describing coals of individual producing States, Technical Paper 269, Analyses of Iowa Coals, has been brought up to date by analyses made since its publication in 1921, and the revised and expanded material was published.<sup>10/</sup> The geology of the coal fields, methods of mining, preparation, production, distribution, uses, and the relationship of mine samples to commercial shipments were discussed. Descriptions of mine, tippie, and delivered samples were given, and chemical analyses, calorific values, classifications by rank, agglomerating indexes, and fusing temperatures of ash were included. The locations of operating mines were included, with data on the nature of roof and floor and on thickness of coal and partings in the beds.

Proximate or ultimate analyses were reported on 10,100 samples from purchases of coal and tippie and breaker inspections taken in connection with Federal fuel purchases, an increase of 47 percent over the previous year. Fusing-temperature determinations were reported on 2,980 of the samples.

From 1,713 inspections of coal mines by the Bureau's coal-mine inspectors, a total of 7,984 samples of road, roof, rib, and gob dust was received and analyzed. These samples, usually received, analyzed, and reported on the same day, guide the inspectors in determining if enough rock dusting is being done in mines to insure safe operation.

The total of 24,681 samples received was not only the largest number ever handled by the laboratory, exceeding the previous year's record number by 565, or 2.3 percent, but, in addition, the actual analytical work involved was even greater because of a large increase in the number of determinations per sample, since more ultimate and proximate analyses were required by more rigid standards in coal purchasing and in connection with foreign cooperative projects; the work-load increase was 9.0 percent.

The averages and the ranges of as-received B.t.u. values of American coals on a national and also on a State basis were determined.<sup>11/</sup> Table 1 gives the national averages:

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- <sup>9/</sup> Snyder, N. H., Handbook on Coal Sampling: Bureau of Mines Handbook, 1950, 10 pp. (Revision of Tech. Paper 133, Directions for Sampling Coal for Shipment or Delivery, by G. S. Pope, revised by N. H. Snyder.)
- <sup>10/</sup> Averitt, Paul, Toenges, Albert L., Olin, H. L., Bell, C. H., Snyder, N. H., Cooper, E. M., Abernethy, R. F., and Tarpley, E. C., Analyses of Iowa Coals: Tech. Paper 706, 1949, 65 pp.
- <sup>11/</sup> Flynn, George J., Jr., Average Heating Values of American Coals by Rank and by States: Bureau of Mines Inf. Circ. 7538, 1949, 11 pp.

TABLE 1. - Average B.t.u. values of American coals

Rank	As-received B.t.u. content, average
Anthracite (Pennsylvania only) .....	12,750
Bituminous (all) .....	13,100
Low-volatile bituminous .....	14,200
Medium-volatile bituminous .....	14,000
High-volatile bituminous .....	12,900
Subbituminous .....	9,550
Lignite .....	7,000
All ranks .....	13,000

A chapter covering sampling, analysis, and properties of coal and other solid fuels was prepared for the twelfth edition of Kent's Mechanical Engineers' Handbook.<sup>12/</sup> Methods of sampling coal and coke, methods of preparing and analyzing laboratory samples, properties of coal and of coal ash, coal specifications, bulk density and miscellaneous related data, and typical analyses of coals from large mines actively in production in 1948-49 were described. Properties and analyses of coke, wood, hogged fuels, and miscellaneous solid fuels were also included.

A number of samples of coals from the United States and from European countries were analyzed in connection with the International Coal Classification Program, wherein there was an interchange of samples among the national coal laboratories of most of the coal-producing countries as part of a program to develop an international standard coal-classification system. Cooperation was extended to the Economic Cooperation Administration through analysis of Greek and Korean coals.

#### Analyses of Miscellaneous Materials

To determine whether certain dusts were suitable for rock dusting in coal mines to prevent coal-dust explosions, 10 samples of limestone, marl, boiler fly ash, etc., were chemically analyzed; 3 were found satisfactory as submitted, and 2 would be if ground finer. A proprietary fuel-saver and 11 other miscellaneous compounds were analyzed to determine their chemical nature. The chemical constituents of 75 coal ash, coal-ash slag, and boiler-water and tube-deposit samples were determined to aid the Bureau's research programs and assist in making recommendations for better operation of boilers and better feed-water treatment.

#### Constitution, Properties, and Analytical Methods

##### Coal Classification

A review and discussion of coal classification by rank, grade, and type was published.<sup>13/</sup>

- <sup>12/</sup> Carnon, E. P., Corey, R. C., Morgan, R. E., and Brewer, R. E., Solid Fuels: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-17 to 2-45, John Wiley & Sons, Inc., New York, N. Y.
- <sup>13/</sup> Carnon, E. P., Solid Fuels. 6. Coal Classification: Kent's Mechanical Engineers' Handbook, Power vol., 12th ed., 1950, pp. 2-17 to 2-20, John Wiley & Sons, Inc., New York, N. Y.

## Petrographic Studies of Coal

The Georges Creek and Upper Potomac coal beds, Allegany and Garrett Counties, Md., were investigated by diamond-drill-hole testing and laboratory study of recovered cores.<sup>14/</sup> All cores from the test holes were studied megascopically and selectively sampled for special analyses and tests. Cores of six commercially important beds studied revealed bright, banded coals having numerous thin lenses of mineralized fusain and pyritic impurities. Thin sheets of mineral impurities, such as calcite and kaolinite, were common to the vertical fracture surfaces. Figure 1 illustrates graphically the character of typical sections of the commercially important coal beds - Barton, Marien, Lower Freeport, Upper Kittanning, Middle Kittanning, and Lower Kittanning.

These observations indicate that much of the ash-forming impurities in certain coal beds consist of mineral matter associated with the coal in form of layers, lenses, and veinlets. The extraneous mineral impurities, which are of much higher specific gravity than the coal, can be removed from the coal by gravity-separation methods of coal cleaning, thereby improving the quality of the coal for combustion and coking uses.

## Electron Microscopy of Coal

A technique has been developed for utilizing the electron microscope in studies of coal microstructures.<sup>15/</sup> The high resolving power of this instrument will be of value in studies of variations in coal microfossils that are found in different beds and areas. These differences in species are used as a basis for identification and correlation of different coal beds in surveys of national coal resources.

The usual thin-section or "peel" methods of coal petrography are inadequate for electron microscopy. In the new technique, the coal specimens are polished by the usual methods, then etched for a short period in a chromic acid-sulfuric acid etchant. After washing and drying, a double film of Formvar (polyvinyl formal)-nitrocellulose is formed on the surface by successively applying solutions of these resins. The film is stripped from the coal surface, specimen screens are fastened over selected areas, and the nitrocellulose is dissolved in acyl acetate. The remaining Formvar film (about 0.1 micron thick), in which the contours and fine details of the specimen surface are reproduced, is observed in the electron microscope.

Figure 2, a sample electron micrograph of coal structures reproduced in such a replica, shows a replica of attrital coal containing microspores, which appear lighter in the micrograph. Many of the details of compression and folding shown here would not be shown clearly by the optical microscope.

<sup>14/</sup> Toenges, Albert L., Turnbull, Louis A., Williams, Lloyd, Smith, H. L., O'Donnell, H. J., Cooper, E. M., Abernethy, R. F., and Waage, Karl M., Investigation of Lower Coal Beds in Georges Creek and North Part of Upper Potomac Basins, Allegany and Garrett Counties, Md.: Reserves, Petrographic and Chemical Characteristics of Coals, and Stratigraphy of Area: Bureau of Mines Tech. Paper 725, 1949, 142 pp.

<sup>15/</sup> McCartney, J. T., Electron Microscopy of Coal: Econ. Geol., vol. 44, November 1949, pp. 617-620.



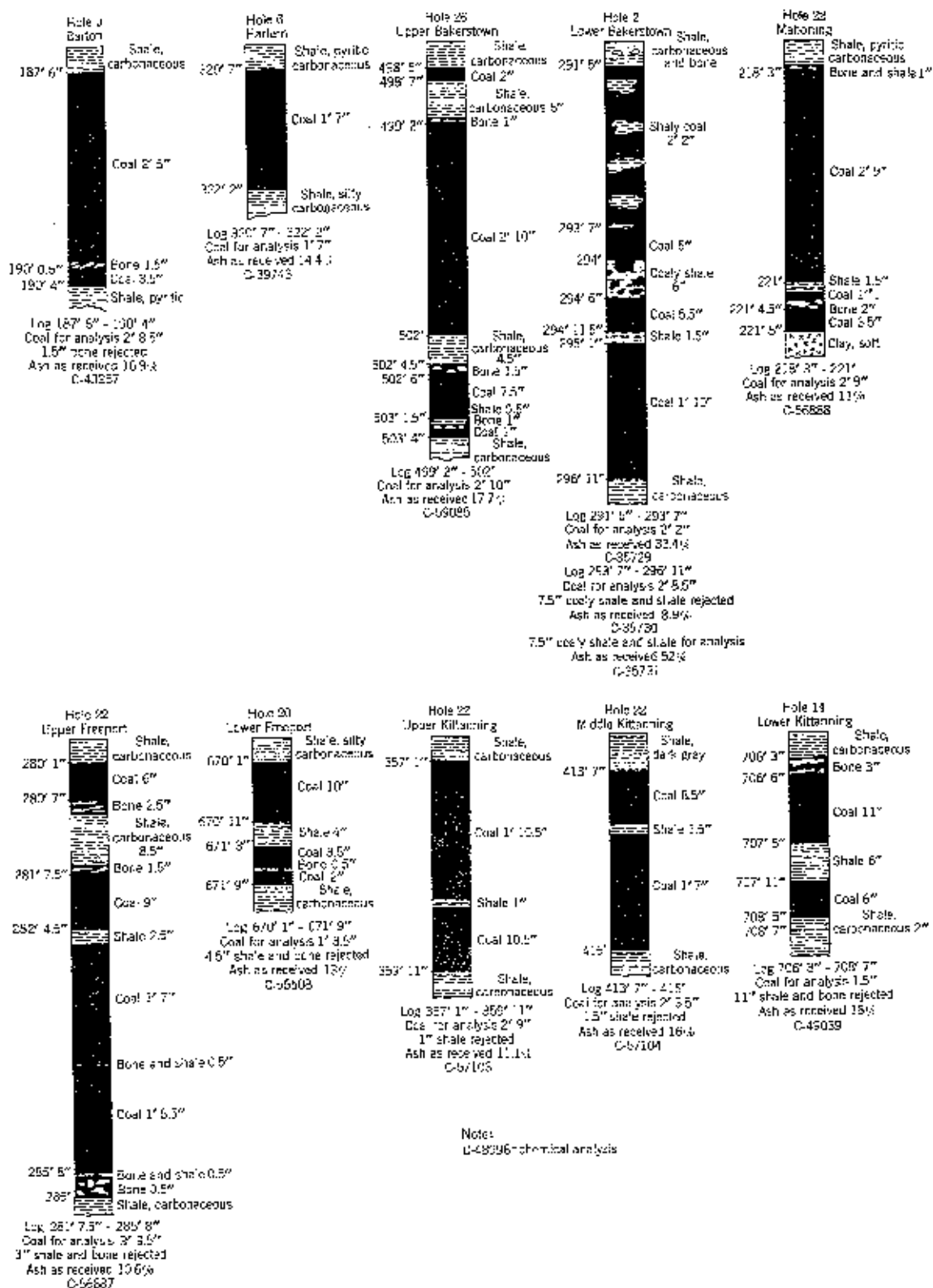


Figure 1. - Typical coal sections of drill cores from Georges Creek and north part of Upper Potomac Basins, Allegany and Garrett Counties, Md.



Figure 2 - Electron micrograph of a Formvar replica of microspores in attrital coal. (X4,500.)

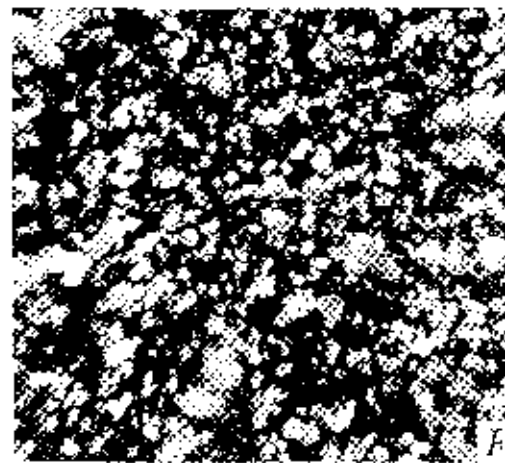
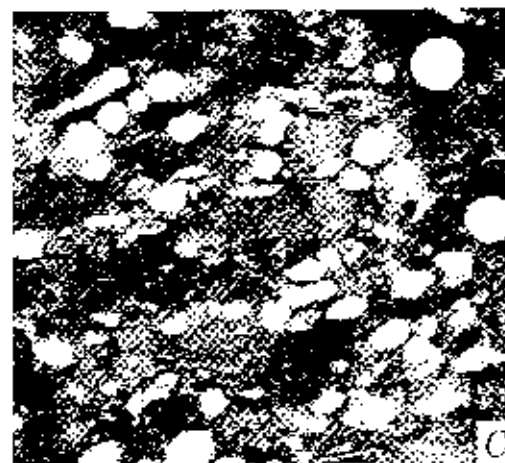
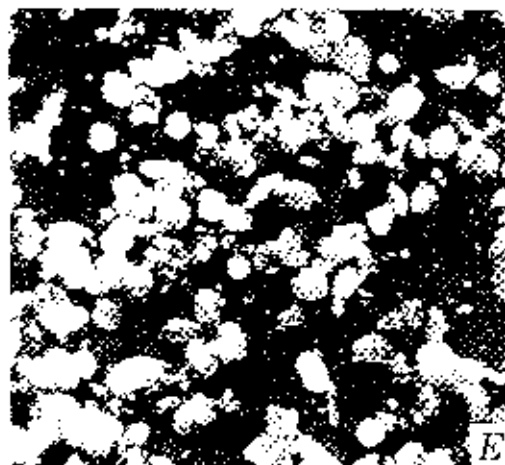


Figure 3. - Photomicrographs of thin sections of lignitic coal and ingredients isolated from attrital lignite; **A**, Coal material derived from woody tissues called anthraxylon and the chief component of xyloid lignite (X200); **B**, Orange-colored humic material, an important component in xylloid and some attrital lignites (X100); **C**, Attrital type lignite containing large, yellow translucent particles (X100); **D**, Resin particles and cuticular material isolated from attrital lignite, plus 180-mesh (X40); **E**, Resin particles of round shape isolated from attrital lignite, 180- x 325-mesh (X67); **F**, Spores, pollen, and fine, yellow granular material from attrital lignite, minus 325-mesh (X100).

## Extractable Waxes from American Lignite

Solvent extraction of certain lignitic coals yields a wax termed "montan wax." The commercial grade of montan wax is a high-melting, hard, brittle wax which has many industrial uses. It is an important ingredient in certain polishes, electrical insulating compositions, leather dressings, inks, carbon papers, protective coatings, greases, and other products. Montan-wax requirements for industry in the United States before World War II were met by imports, nearly all of which came from Germany. Since the war, montan wax has not been available from Germany, but relatively small amounts have been imported from Czechoslovakia.

Because of the shortage of montan wax at the close of the war and the fact that American industry in the past had to rely on foreign sources, a laboratory investigation was started in 1945 to obtain information concerning yields and properties of solvent-extractable wax from domestic lignites. Published results of the investigation<sup>16/17</sup> include discussions of the occurrence and geology of lignite deposits of the United States, from which samples were obtained for wax extraction, and the petrographic composition of lignites, particularly in respect to those components that contribute to wax yields.

The survey of the solvent-extraction yields showed that the highest yields were obtained from certain Arkansas and California lignites. Some of these yields, particularly those obtained with a mixture of benzene and alcohol, were similar to those obtained in commercial extraction of montan wax from German brown coal. The lignites examined from Montana, North Dakota, Texas, and Washington gave appreciably smaller quantities of solvent extract than the Arkansas and California lignites.

Although higher yields of extract were obtained with a solvent mixture of benzene and alcohol than with benzene alone, the properties of the benzene extracts more closely resembled commercial grades of imported montan wax than did the benzene-alcohol extracts. The most significant difference between the extracts obtained in this investigation and the Ricbeck brand of montan wax from Germany was the greater resin content of the extracts from domestic lignites; they more closely resembled a commercial grade of montan wax from Czechoslovakia.

A study of the petrographic composition of American lignites showed that the high wax-yielding lignites are attrital and consist predominately of plant remains of small particle size, called attritus, much of which is yellow translucent matter under the microscope (fig. 3, C). Woody or xylloid lignites contain only small quantities of extractable wax; they consist largely of orange, translucent coal material of relatively large particle size, which was derived mainly from wood tissues of plants (fig. 3, A and B).

The main source of the soluble material extracted from lignite, which consists of wax, resin, and asphaltic material, was certain ingredients in the attrital ground mass. These ingredients were yellow cuticular material, round resinous bodies, lump-type resinous particles, spores, pollen, and very fine, yellow granular matter of uncertain origin (fig. 3, D, E, and F). Cuticular material was undoubtedly the original source of much of the waxy fraction.

16/ Selvig, W. A., Ode, W. H., Parks, B. C., and O'Donnell, E. J., American Lignite: Geological Occurrence, Petrographic Composition, and Extractable Waxes: Bureau of Mines Bull. 492, 1950, 63 pp.

17/ Ode, W. H., and Selvig, W. A., Extractable Waxes from American Lignites: Ind. Eng. Chem., vol. 42, January 1950, pp. 131-135.