REVIEW OF BUREAU OF MINES COAL PROGRAM, 1969

By John D. Spencer

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REVIEW OF BUREAU OF MINES COAL PROGRAM, 1969[1]

by

John D. Spencer[2]

INTRODUCTION

Projects aimed at achieving the least disturbance to the Nation's air, water, and land resources in the mining and utilization of coal continued to be a feature of the Bureau of Mines coal program. This reflects the current concern about damage inflicted on the environment by our growing and increasingly affluent society.

Mine Health and Safety research, along with investigations related to all other aspects of coal extraction and use, also continued to be implemented and expanded.

The overall research efforts seek the dual objective of increasing coal tonnage yet facilitating pollution abatement, and control in achieving maximum public benefit from our coal resources.

Appendix A lists the Bureau of Mines research centers and laboratories. Publications issued during 1969 are cited with the projects and in appendix C. Organizations that cooperated with the Bureau in the work described in this report are cited in appendix B.

Trade names appearing in this report are used to facilitate understanding and do not imply product endorsement by the Bureau.

SECTION 1. - ENVIRONMENTAL ACTIVITIES

Mined-Land Rehabilitation

Surface Reclamation

Two surface mine reclamation projects under the Appalachian Regional Development Act were completed in 1969. In the Delano project, Schuylkill

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[1] The 34th in a series. Previous report: Spencer, John D. Review of Bureau of Mines Coal Program, 1968. BuMines Inf. Circ. 8416, 1969, 94 pp.

[2] Chemical engineer, Morgantown Coal Research Center, Bureau of Mines, Morgantown, W. Va.

County, Pa., old abandoned strip pits were backfilled, and the surface was graded for development of an industrial park. Since completion of the work, considerable interest has been aroused for building sites in the park. Eventually, a number of job opportunities will be available in the area.

The Pittsburgh, Pa., Airport project also was a strip-mined area. Exposed highwalls along the landing strips created a safety hazard to aircraft using the airport. Backfilling and grading of the mined area eliminated the highwalls, and provided a safer approach to runways for all aircraft.

Two additional mine reclamation projects, one in Jefferson County, Ohio, and one in Wise County, Va., were approved by the Secretary of the Interior. The Ohio project is part of a 1,100-acre tract of land to be developed into a large recreational area known as Friendship Park. Project work is to consist of building an earth-filled dam to create a 90-acre lake, and backfilling, grading, and seeding of strip-mined land adjacent to the lake. When completed, the park will provide recreational facilities for a large population in eastern Ohio and northern West Virginia. The Virginia project consists of backfilling abandoned strip pits, sealing old underground mine openings, and grading and seeding the spoil area. This effort will eliminate a public safety hazard from exposed surface and underground mines and provide a site for new educational facilities for the city of Norton, Va.

Three more subsidence control projects were completed--one in Coaldale, Pa., and two in Wilkes-Barre, Pa.--and two more projects are being implemented in Scranton, Pa. Total contract cost of these and the two projects previously completed is estimated at almost $6 million. The significance of these projects is that they will afford protection to about 200,000 people and property valued at about $112 million..

Four other projects, to backfill abandoned mine voids to protect public health and safety, have been approved by the Secretary of the Interior. Total contracted cost of these projects is almost $1,500,000. Subsidence control under the Appalachian program is also being continued.

Mine-Fire Control

Fire Control Projects

Thirty-four mine-fire-control projects have been approved under the Appalachian Regional Development Act of 1965, at a total estimated Federal cost of $16,800,000. A summary of project status follows:

Status	Anthracite	Bituminous
Completed before 1969...........	0	10
Completed in 1969..............	1	5
In progress, 1/1/70............	6	2
Total......................	7	17

One anthracite project is being completed under total State supervision and funding, and the State has also withdrawn its application for Federal assistance in connection with another project previously approved by the Appalachian Regional Commission. At the end of 1969, the eight remaining projects were being processed toward the actual work stage.

Eight new fires in the Appalachian bituminous coalfields in two States were investigated in cooperation with local officials. Full responsibility and funding for control work on two of these fires has been undertaken by State authorities, while action is pending by State and local officials toward submitting proposals to the Appalachian Regional Commission for controlling five of the other fires. The eighth fire, which was small and under very thin overburden, was extinguished by the property owner.

Mine fires outside the Appalachian States are controlled under a Public Law (83-738) that provides for equal sharing of control costs by the Federal Government and the cooperating State or private property owner when the fire is on other than Federal or Indian land. In the latter case, or where Federal coal is threatened by a fire on private land, the full cost is defrayed by the Government. During 1969, six control projects were initiated and completed in Western States in federally owned coalfields--three in Colorado, two in Utah, and one in Wyoming. In addition, the second phase of control work on a major coalbed fire in Utah was completed. By the end of the year, 190 fire-control projects had been completed at a Federal cost of almost $5 million, conserving an estimated 762 million tons of coal.

Fire Control With Fly Ash

Injection of dry fly ash through boreholes to control fires in abandoned underground coal mines has been used in six bituminous and one anthracite project since inception of the technique in 1967. More recently, an innovation in the technique was introduced in three of the projects. In the innovation, water was mixed with the ash and injected in the form of a slurry, following maximum pneumatic injection of dry fly ash. Although data on the efficiency of dry ash versus slurry injection are unavailable, field experience indicates that slurry injection provides a satisfactory barrier in special applications. In one project, dry fly ash was injected to the point of refusal through boreholes into an underground area to form a barrier to halt the advance of a mine fire. A slurry consisting of 70 weight-percent of fly ash was then injected under pressure in the same area to assure protection of private homes. Despite the previous injection of fly ash to the point of refusal, significant additional quantities of fly ash slurry went in, indicating that pressure injection of slurry more adequately filled the minute spaces within the ash.

Research on underground void filling with dry fly ash was extended in experiments using fly ash-cement-water grouts to fill and seal caved areas. Fly ash grouts containing up to 10 percent Portland cement and 100 gallons of water per ton of solid were pumped hydraulically into simulated rubble-filled caved areas. Flow characteristics were good, and airtight seals were obtained which served as effective roof support. The method has now been applied in more than a dozen areas in bituminous and anthracite coalfields where underground fires or subsidences created serious problems.

Infrared Sensing of Coalbed Fires

Encouraged by initial results of investigations of aerial infrared sensing to detect and delineate underground mine fires, a pilot project was begun in the western coalfields in 1969. Because some coalbed fires are in remote and sometimes almost inaccessible locations, an efficient aerial-sensing system would greatly reduce the time and cost of physically surveying the coalfields to detect new fires and delineate the advance of known fires.

Results of the initial infrared sensing flights in a 600-square-mile area of coalfields in Arizona, Colorado, New Mexico, and Utah indicate that the technique can be used to successfully detect outcrop fires or fires under shallow overburden. Imagery produced by the infrared sensor correlated well with the information obtained by ground checks of known fires. The sensor also detected four unknown fires in the test area.

Microwave sensing as a means of detecting voids created by mine fires was investigated in two areas in cooperation with the U.S. Geological Survey to determine the applicability of this technique in discovering and delineating underground fires. Evaluation of the results was begun.

Acid Mine-Water Drainage Control

Anthracite mine-water-control efforts were continued. Of 37 projects approved by the Secretary of the Interior before 1969, four were canceled before any public funds were spent because the mines operating in the reserves to be protected were shut down for economic or other reasons. Contracts were signed calling for equal contribution of money by the Federal and State governments to cover the full costs of the facilities. Total outlay was about $7,500,000.

In 17 of the projects, 29 large pumps were used to control the levels of underground mine-water pools that otherwise would endanger active mines. Fourteen of the pumps were installed at a total cost of $5,630,000. Four pump projects involving the exchange or transfer of equipment were completed at no cost to the Federal Government. Four of the 29 pumps were supplied by the Commonwealth of Pennsylvania; only the installation costs were charged to the program.

Sixteen surface drainage control systems were installed, at a contracted cost of almost $2 million, to prevent approximately 3.5 billion gallons of water from seeping into or flooding underlying mine workings each year.

Drainage Control in Operating Mines

Monitoring at the Conemaugh mine, North American Coal Company, Seward, Pa., showed the following quality and quantity:

	North section	Total discharge
Average.............gpd..	100,000	450,000
Total acidity........ppm..	500	2,500
Iron................ppm..	175	1,350

Air samples from Hixson No. 4 borehole in the north end of the north section decreased in oxygen content from 7 to 5 percent between January and July 1969. Channel samples collected at widely spaced locations in the mine indicate a high pyritic sulfur content in the coal and immediate roof rock; the highest average value for the coalbed was 5.7 percent.

A core-drilling program at the Warwick No. 3 mine, Duquesne Light Company, Greensboro, Pa., promises to yield more detailed information on stratigraphy and ground water in the overburden than was previously available. The holes are being drilled to prove coal reserves in advance of mining.

Mine Sealing

The mine-air sealing experiment at the Decker No. 3 mine, Kittanning, Pa., was concluded in mid-1969. A reduction to 17 percent in the average oxygen content of the mine atmosphere did not significantly change the gradually decreasing trend of the total acidity of the mine effluent, although a minor decrease in acid load was attributed to sealing. It was concluded that air sealing was not more effective in reducing the oxygen in the mine because of the fractured shallow overburden and depressed water table.

Bulkheads were constructed in the main entries of United States Steel Corporation's worked-out Karen mine, Centerville, Pa. Also, the remaining portal was sealed by erecting an exterior steel dam to waterflood the portal area to the roof. Proposed flooding of the mine is illustrated in figure 1.

Mine-air samples from four surface drill holes showed widely differing oxygen concentrations, which have ranged from 0.05 to 20.43 percent. Continuous monitoring of oxygen levels and differential pressures at a selected drill hole correlate with changes in atmospheric pressure and indicate "mine breathing" through the overburden. The mine effluent remains virtually free of acidity, but high in iron and sulfate, indicating neutralization by alkaline ground water in the mine.

Mine Water Treatment

Plant design and cost estimates have been developed for limestone treatment of acid mine water for a wide range of water rates and compositions. The treatment (fig. 2) consists of limestone neutralization, aeration, solids settling, and concentration. The design criteria for the various processing steps are based on pilot-plant data,[3] laboratory data, and mine-water

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[3] Mihok, E. A., Maurice Deul, D. C. Chamberlain, and J. G. Selmeczi. Mine Water Research: The Limestone Neutralization Process. BuMines Rept. of Inv. 7191, 1968, 20 pp.

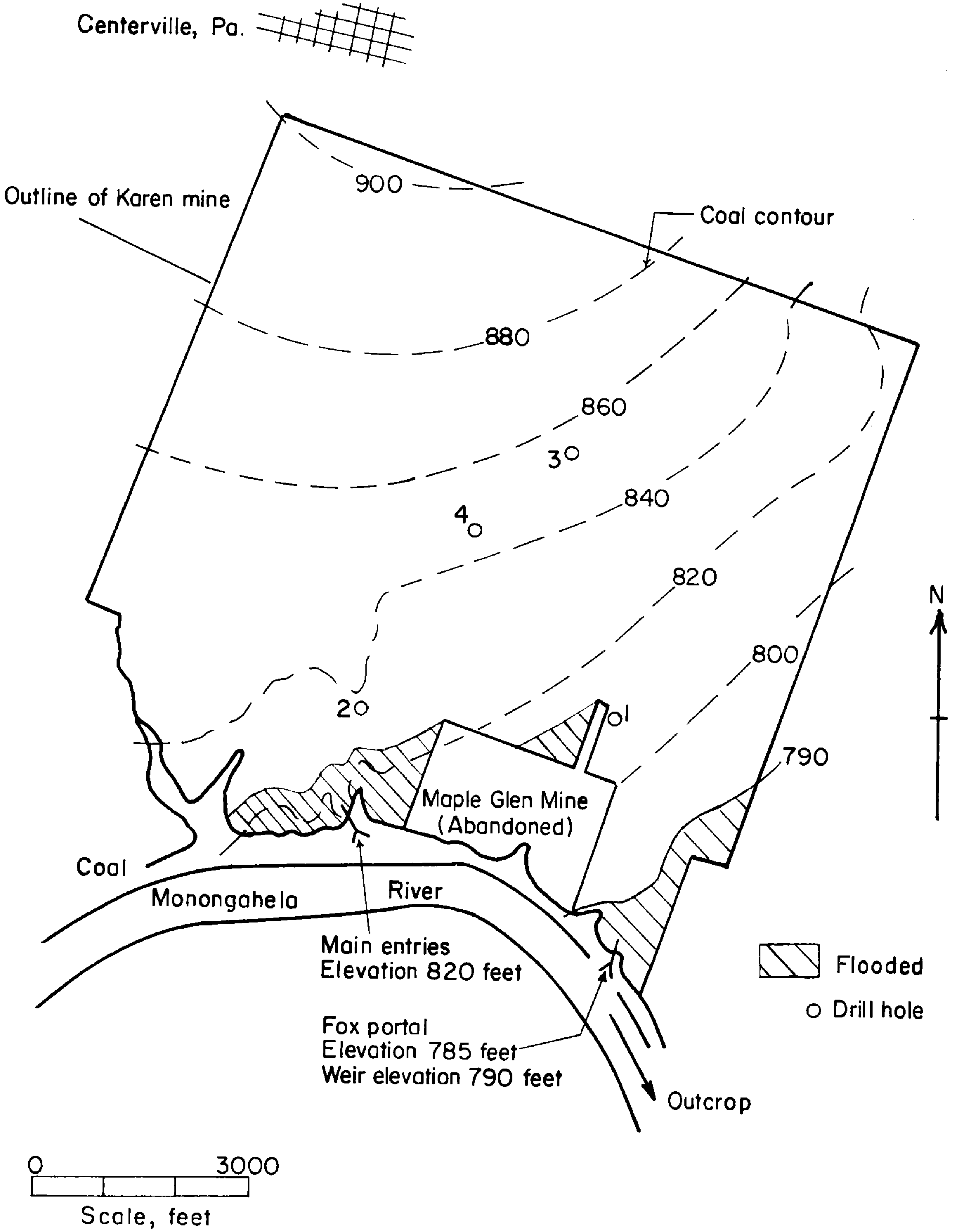

FIGURE 1. - Flooded Sections of Karen Mine, Centerville, Pa.

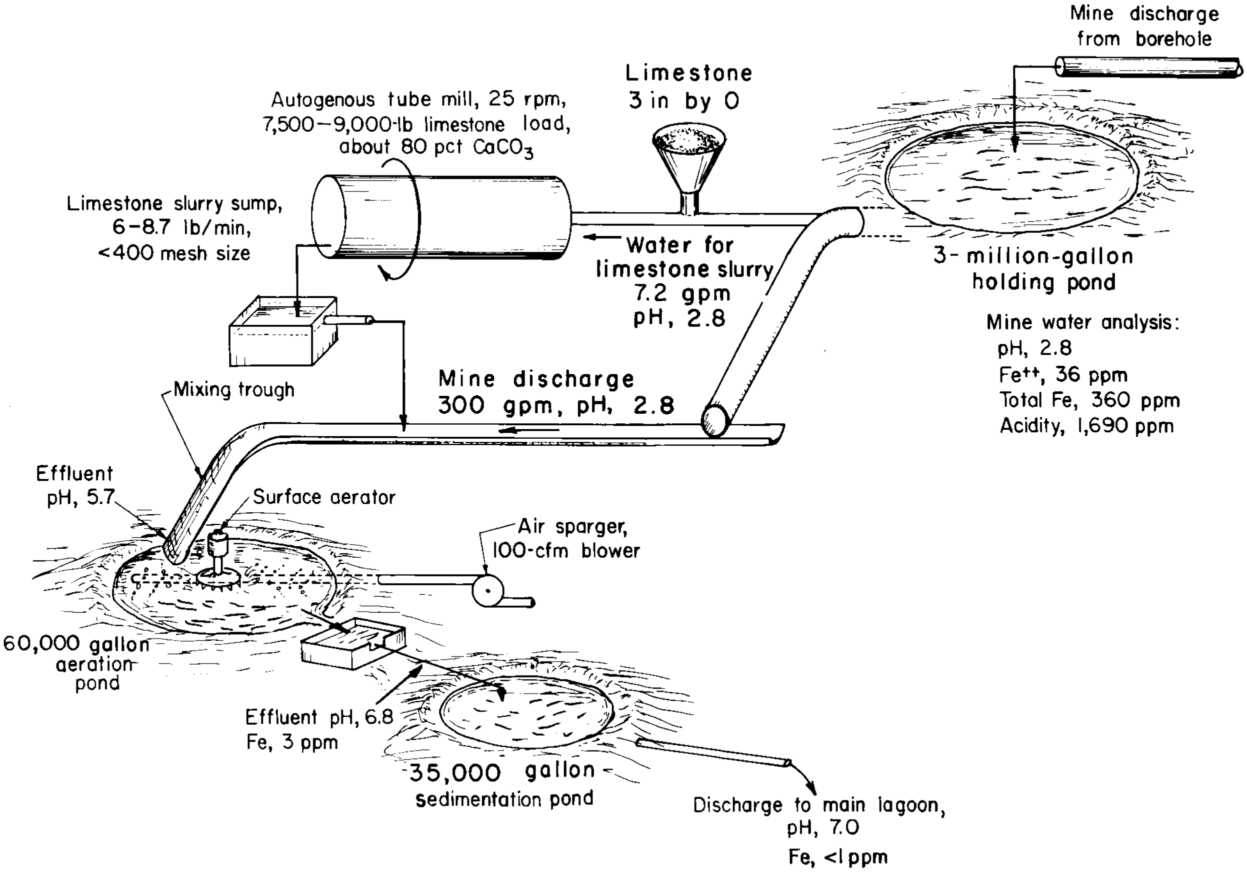

FIGURE 2. - Flowsheet of the Mine Water Neutralization Process.

treatment standards. Unit costs have been assumed and are related to general market prices and conditions. These estimates serve as preliminary indicators of the cost of constructing and operating a mine water treatment plant. Two factors directly connected with mine water treatment, but not considered because of the extreme variability reflected by site conditions, are land costs and removal of the water from the mines. For these cost estimates, sufficient land is assumed to be available, and mine water is discharged into holding ponds.

While the engineering costs for the limestone treatment process, including process optimization and simplification, were being developed, the concept of catalytic oxidation of acid mine water with activated carbon was tested.[4] In laboratory batch-flow tests on a highly ferriginous acid mine water, activated carbon reduced the ferrous iron content from about 700 to about 10 parts per million in less than 1 minute. Moreover, the carbon was used repeatedly with little loss in efficiency or solids deposition. Promising aspects of the

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[4] Mihok, Edward A. Mine Water Research: Catalytic Oxidation of Ferrous Iron in Acid Mine Water by Activated Carbon. BuMines Rept. of Inv. 7337, 1969, 7 pp.

activated carbon treatment are (1) nonrecurring expense; and (2) long life of the carbon. With all the iron in mine water converted to the ferric state, neutralization by limestone can be effected almost instantaneously, and subsequent aeration is unnecessary or minimal. This should greatly simplify the neutralization process and reduce treatment costs almost to an irreducible minimum.

Extinguishing Coal-Refuse Banks

A demonstration project was initiated in Pennsylvania to extinguish a burning coal-refuse bank, with special techniques employed to minimize gas and dust output and explosions during the extinguishment process. A series of earthen dikes were constructed atop the burning pile to form impoundments, subsequently flooded with water pumped from an underlying, abandoned mine. This water seeped through the burning mass, cooling it sufficiently to permit transfer to a new disposal area following additional quenching and cooling. At the new disposal site, the refuse was graded to the desired contour, compacted, and covered with a layer of noncombustible earth. An important phase of the project involved setting up instruments to monitor the atmosphere. Data were obtained concerning the types and concentration of gases and dusts emitted from burning refuse banks.

Solid Waste Disposal and Utilization

Scrap Rubber

Results were published of cooperative research with the Firestone Tire and Rubber Company on the carbonization (destructive distillation) of scrap tires.[5] Carbonization was demonstrated to be a technically feasible method of disposing of and obtaining potentially valuable products from scrap automobile and truck tires. Amounts of various products were shown to be highly dependent on carbonization temperature. Highest yields of liquid products were obtained at 500° C (930° F); highest yields of solid and gaseous products were obtained at 900° C (1,650° F). Light oils produced at the higher temperature contained substantial amounts of benzene and toluene, whereas those from the lower temperature tests contained more highly saturated hydrocarbons. Residues from all tests were friable carbonaceous materials. Heating value of gases ranged from 890 to 922 Btu/cu ft, at 500° C, to 693 to 765 Btu/cu ft at 900° C, which approaches the heating value of natural gas--about 1,000 Btu/cu ft.

Municipal Refuse

Research was actively pushed to establish the prospects and problems in processing urban wastes into oils that may serve as a source of fuels and chemicals. Ready conversion of cellulose with $CO + H_2O$ in hydrogenation and hydrocracking experiments led to successful tests in which municipal refuse, which is largely cellulosic in content, was transformed into oil. About 90

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[5] Wolfson, D. C., J. A. Beckman, J. G. Walters, and D. J. Bennett. Destructive Distillation of Scrap Tires. BuMines Rept. of Inv. 7302, 1969, 19 pp.

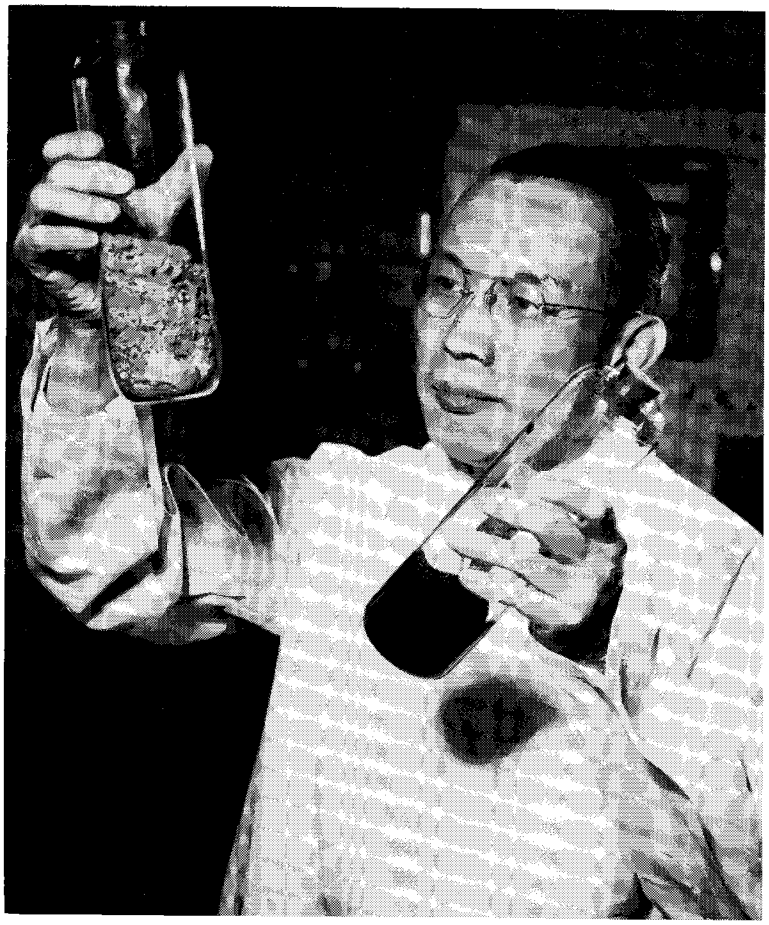

FIGURE 3. - Municipal Refuse Charge (Left); Oily Hydrogenation Product (Right).

percent of the refuse was converted into oil, amounting to approximately 40 weight-percent of the original refuse (dry and ash-free basis). Exploratory experiments were also conducted with refuse (from Altoona, Pa.) to determine if such material is amenable to conversion to oil by hydrogenation. In one test, at 4,000 psig and 800° F, and without a catalyst, 34 percent of the moisture- and ash-free refuse charged (fig. 3) was converted into a benzene-soluble oil. Benzene-soluble tar produced by the hydrogenation of the same refuse was found to contain saturated hydrocarbons and oxygenates; stearic and palmitic acids were specifically identified. The tar consisted mostly of saturated material, with hydroxyl groups and small amounts of phenols, acids, ethers, and olefins also present. Neutral oils from the carbonization of industrial plant refuse contained predominantly alkyl benzenes, alkyl naphthalenes, styrenes, indenes, and indans. Average molecular weights of the products varied inversely with the carbonization temperature. The tar acid fraction contained approximately 70 percent alkyl phenols.

An incinerator for combustible wastes (fig. 4) was built and tests were begun with mechanical feeding equipment for the unit. The incinerator is designed to burn 100 pounds of refuse per hour, but preliminary tests indicate higher combustion rates may be possible. Difficulty was experienced with the screw-feeding mechanism and feed hopper and several design changes were made and tested. Combustion tests will be made with standard refuse materials, paper, cardboard, and potatoes to establish the best operating conditions for the incinerator, after which actual municipal refuse will be processed.

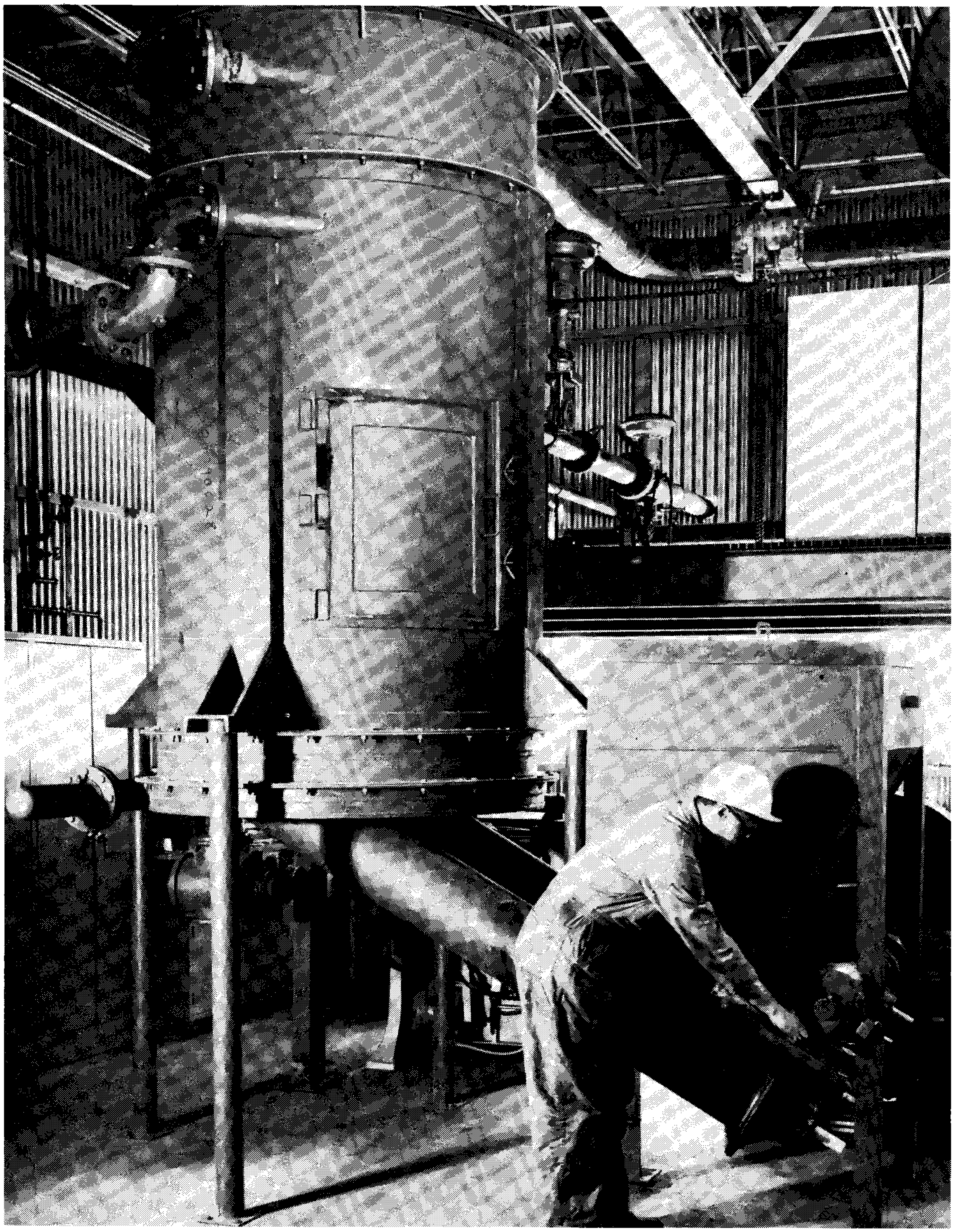

FIGURE 4. - Combustible Waste Incinerator.

Coal Wastes

Sources and Disposal Methods

A study of coal waste disposal methods at a coal mine in Kentucky and another in Alabama was completed to develop cost estimates, provide better knowledge of current disposal practices, and guide consideration of alternate disposal methods.[6] Although total disposal costs were essentially the same for both operations, reclamation costs were found to vary because of different reclamation practices.

A report was released describing solid waste disposal problems in the Pennsylvania anthracite region.[7] Located and identified were 863 banks containing more than 900 million cubic yards of material occupying a total combined area of almost 20 square miles. Most of the refuse piles are located within 2 miles of traffic centers of incorporated communities.

The history of coal mining in the Pacific Northwest was related to past and future generation of and disposal of coal refuse resulting therefrom.[8]

A comprehensive examination was completed of all known burning coal refuse banks. Work was begun on a report that will describe the location and size of each bank, the impact of smouldering piles on the environment, and various techniques for extinguishing burning banks.

Coal and Coal Waste Analysis

Minerals in coal ash and related materials are being identified and classified by modern analytical techniques to facilitate development of processes for the utilization of coal and coal waste. For example, advances have been made in determining the potential of far-infrared spectroscopy for identifying coal-related minerals. Bureau researchers obtained far-infrared spectra for 18 different minerals and inorganic materials (fig. 5) and used the information to identify calcite and dolomite in coal refuse, dawsonite and dolomite in oil shale, thenardite in a boiler deposit, and ferrous sulfate in the surface film on pyrite from waste coal.[9] Identification of these substances represents original applications of far-infrared spectroscopy for analytical purposes.

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[6] Danielson, V. A., and D. H. White, Jr. Waste Disposal Costs at Two Coal Mines in Kentucky and Alabama. BuMines Inf. Circ. 8406, 1969, 28 pp.

[7] MacCartney, John D., and Ralph H. Whaite. Pennsylvania Anthracite Refuse, A Survey of Solid Waste From Mining and Preparation. BuMines Inf. Circ. 8409, 1969, 77 pp.

[8] Geer, Max R. Disposal of Solid Waste From Coal Mining in Washington, Oregon, and Montana. BuMines Inf. Circ. 8430, 1969, 39 pp.

[9] Karr, Clarence, Jr., and John J. Kovach. Far-Infrared Spectroscopy of Minerals and Inorganics. Appl. Spectry., v. 23, No. 3, May-June 1969, pp. 219-223.

FIGURE 5. - Identifying Minerals in Coal Refuse by Far-Infrared Spectroscopy.

Another advanced technique, nuclear quadrupole resonance (NQR) spectrometry, was used, possibly for the first time, for quantitative analyses of mineral mixtures and natural ores.[10] Calibration curves for several minerals were obtained from signals from the copper-63, copper-65, chlorine-35, and bismuth-209 isotopes. These curves were used to analyze several samples of cuprite ore for cuprous oxide content.

Low-frequency infrared spectroscopy was shown to have unique capabilities for the characterization and analysis of minerals in coal and other materials. Infrared spectra and analyses were obtained for a variety of iron minerals that occur in bituminous coals, including sulfides, carbonates, oxides, hydrous oxides, and sulfates.[11] Several useful spectral distinctions, unique for coal minerals analysis, were also pointed out.

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[10] Schultz, Harry D., and Clarence Karr, Jr. Quantitative Aspects of Nuclear Quadrupole Resonance Spectrometry of Inorganics and Minerals. Anal. Chem., v. 41, No. 4, April 1969, pp. 661-664.

[11] Estep, Patricia A., John J. Kovach, Clarence Karr, Jr., Edward E. Childers, and Arthur L. Hiser. Characterization of Iron Minerals in Coal by Low-Frequency Infrared Spectroscopy. Preprints, Div. Fuel Chemistry, Am. Chem. Soc., v. 13, No. 1, Apr. 13-18, 1969, pp. 18-34.

The Bureau's expertise in advanced and original spectroscopic techniques for characterization and analysis of minerals was recognized through awards of research contracts by the National Air Pollution Control Administration (NAPCA) to characterize mineral pollutants during fiscal year 1970. Similar recognition was evidenced by the National Aeronautics and Space Administration's (NASA) selection of the Bureau to analyze during 1970 lunar samples from Apollo Missions 12 and 13.

Research was conducted on different kinds of sulfur bondings representative of sulfur modes in the organic portion of various coals, using a novel pulsed nuclear magnetic resonance (NMR) spectrometer that detects signals from the sulfur-33 isotope. Signals could be obtained from concentrations of the NMR-active sulfur-33 isotope as low as 0.007 percent. Model sulfur compounds among which a variety of sulfur modes were detected and distinguished include carbon disulfide, sulfuric acid, dimethylsulfoxide, 1,2-ethanedithiol, and 2,5-dimethylthiophene. Part of this research was conducted under a National Research Council-BuMines Postdoctoral Research Associateship for fiscal year 1970.

Fly Ash

Reclamation of surface-mine spoil with fly ash continued to be investigated at two sites in northern West Virginia. At one of the sites, following application of fertilizer at higher rates, hay production from fly-ash-reclaimed spoil was 4.3 tons per acre, nearly double the 1968 yield (fig. 6). By comparison, the average hay yield for West Virginia in 1969 was 1.5 tons per acre. Nearby, in cooperation with the U.S. Forest and Soil Conservation Services, efforts are being made to utilize mechanical stabilization to reduce land erosion and stream siltation from surface-mine spoil (fig. 7). The method appears to have promise.

At the second site, a cooperative study is underway with the U.S. Forest Service to determine survival rates and growth of various species of trees on fly-ash-treated spoil. Thus far, it appears that pines grow better on such soil than do leafy species. Included in this study is an investigation of moisture penetration and retention in fly-ash-treated spoil (fig. 8).

Evaluation of fly ash as a source of plant nutrients for agricultural soils was continued by Virginia Polytechnic Institute, Blacksburg, Va.

In laboratory and greenhouse experiments, certain fly ashes were found to increase the amount of boron and molybdenum available to alfalfa grown in deficient soils. Fly ash application to soils also increased the zinc and potassium uptake by corn plants. Field studies of a similar nature are planned for 1970.

Investigation was completed of the application of sintered fly ash to beneficiate turf on golf courses, athletic fields, public parks, and similar

FIGURE 6. - Cows Grazing on Surface-Mine Spoil Reclaimed With Powerplant Ash.

areas.[12] Addition of about 30 volume-percent of sintered ash to the top 6 inches of soil increased the water infiltration rate, pore space, root density, and grass yields. These factors contributed to development of a healthy sod by improving soil drainage, aeration, and resistance to compaction.

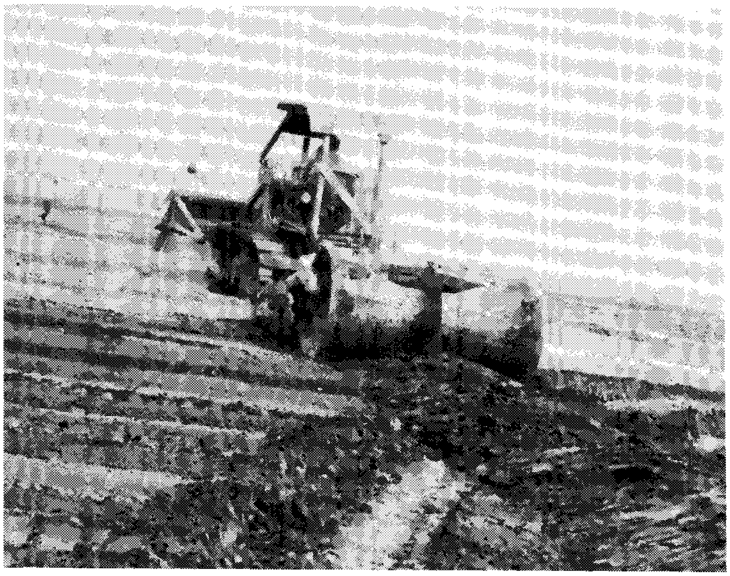

FIGURE 7. - Stabilizing Surface-Mine Spoil To Reduce Erosion and Stream Siltation.

FIGURE 8. - Determining Moisture Penetration of Fly-Ash-Treated Mine Spoil.

Desulfurization of Coal

Centrifugal Separation

Promising results were obtained in the centrifugal (air) separation of pyrite from coal by carefully controlling the airflow to provide a reasonably narrow particle size range to the centrifugal section of the unit. Such control made density, rather than size, the chief factor contributing to particle mass, thus facilitated separation of the heavier pyrite from the coal. Combining this method of particle-size control with coal-grinding operations should improve the efficiency of centrifugal separation. Work was continued.

Following investigation of the application of centrifugation (wet) to separate pyrite and other heavy materials from coal, a

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[12] Patterson, J. C., Jr. Sintered Fly Ash as a Soil Modifier. M.S. Thesis, West Virginia University, Morgantown, W. Va., 1969, 114 pp. (Available from West Virginia University library.)
Patterson, J. C., Jr., P. R. Henderlong, and L. C. Adams. Sintered Fly Ash as a Soil Modifier. Proc. W. Va. Academy of Science, v. 40, 1968, pp. 151-159.

peripherally fed centrifuge was designed for continuous and separate removal of refuse from clean coal. Fabrication of the centrifuge was completed and all operating components have been assembled and installed into a complete operating unit.

Electrical Separation

During the year, certain variations in operating conditions were found to improve the separation of pyrite from fine coal by electrostatic means. Increased peak height (of the pulsed direct current voltage applied to the ionizing field), better cleaning of the grounding electrode, and reduced feed rates all improved the separation efficiency. Pyrite separation efficiency has been limited chiefly by the presence of fine dusty material that prevents proper contact between the charged particles and the grounding electrode. Work was continued to overcome this difficulty.

An electrophoresis column was designed and constructed that utilized not only the different electrophoretic mobilities of coal and pyrite, but also their different specific gravities to separate the two materials (fig. 9). In the column, a sample of coal high in pyritic sulfur was fractionated into three distinct components: siliceous refuse (45 percent ash, 0.4 percent pyritic sulfur), coal (9 percent ash, 0.6 percent pyritic sulfur), and pyritic refuse (45 percent ash, 21 percent pyritic sulfur).

Magnetic Separation

A report is being prepared covering the effect of three grinding actions on the magnetic susceptibility of pyrite. It will include the results from grinding in a ball mill, hammer mill, and disk mill with grinding elements of magnetic and nonmagnetic material.

Grinding To Facilitate Pyrite Removal

Comparative grinding tests of 16- by 30-mesh blends of coal and pyrite showed that a ball mill is much more selective than a ball-and-race-mill during the early stages of grinding. In the latter stages of grinding, however, the situation was reversed. Further work may demonstrate important relations that will help establish the best grinding method for a particular coal.

Organic Sulfur Removal by Microbes

Microorganisms like bacteria, yeasts, and other minute living plants have been under investigation as a means of reducing the organic sulfur content of coals. In recent work, assays were completed for (1) microbially treated and untreated samples of an Illinois No. 5 coal, from which much of the original pyritic sulfur had been removed by flotation, and (2) an Iron County, Utah, coal naturally high in organic sulfur and low in inorganic sulfur. Typical results follow:

	Sulfur content, percent			
	Illinois No. 5 bituminous coal		Utah bituminous coal	
	Untreated	Microbially treated	Untreated	Microbially treated
Sulfur type:				
Organic.......	2.5	2.3-2.6	5.6	5.4-5.8
Inorganic.....	1.2	1.1-1.3	.7	.6- .8

These results do not indicate any microbial effect on the sulfur content of the two coals under the conditions of these tests.

Sulfur Survey of Coal Reserves

Work was continued to identify the steam and coking coal reserves in the Appalachian region by sulfur content, availability, and costs. The primary objective is to determine available quantities of coals of various sulfur-content levels, with special emphasis on lower sulfur coals that may be suitable for heat and power generation in highly industrialized areas regulated as to fuel types to reduce air pollution. Information on this survey is to include f.o.b. mine values, transportation costs, destinations, principal consumer categories, and commitments of reserves.

Studies were initiated to measure the social and economic effects on high-sulfur coal-producing regions caused by shifts in demand to low-sulfur coal-producing areas. Also begun were studies of two metropolitan areas to show the effects, on both consumers and producers of high-sulfur fuels, of changes in the pattern of energy resource supplies resulting from legislation or other regulations on the allowable sulfur content of fuels.

In an effort to correlate the size of pyrite particles and coal-pyrite particle association with the removal of pyrite from coal before burning, a study was conducted of 61 coals presently being used for electric power production.[13] The results were obtained by visual microscopic studies of polished briquets of minus 14-mesh samples. Mean pyrite-particle sizes ranged from 20 to 400 microns, and proportions of pyrite contained in coal particles in amounts greater than 50 percent by volume (estimated) of the particle ranged from 20 to 95 percent. These parameters were correlated with reductions in pyrite accomplished by float-sink tests at 1.60 specific gravity on 14-mesh, 3/8-inch, and 1-1/2-inch samples. The correlation coefficients between pyrite removal and mean pyrite particle size were 0.89, 0.84, and 0.79, respectively, for these samples. Figure 10 shows the relation for the minus 14-mesh samples of 61 coals. Correlation coefficients between pyrite removal and the coal-pyrite association parameter were 0.92, 0.90, and 0.85,

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[13] McCartney, J. T., H. J. O'Donnell, and Sabri Ergun. Pyrite Size Distribution and Coal-Pyrite Particle Association in Steam Coals: Correlation With Pyrite Removal by Float-Sink Methods. BuMines Rept. of Inv. 7231, 1969, 18 pp.

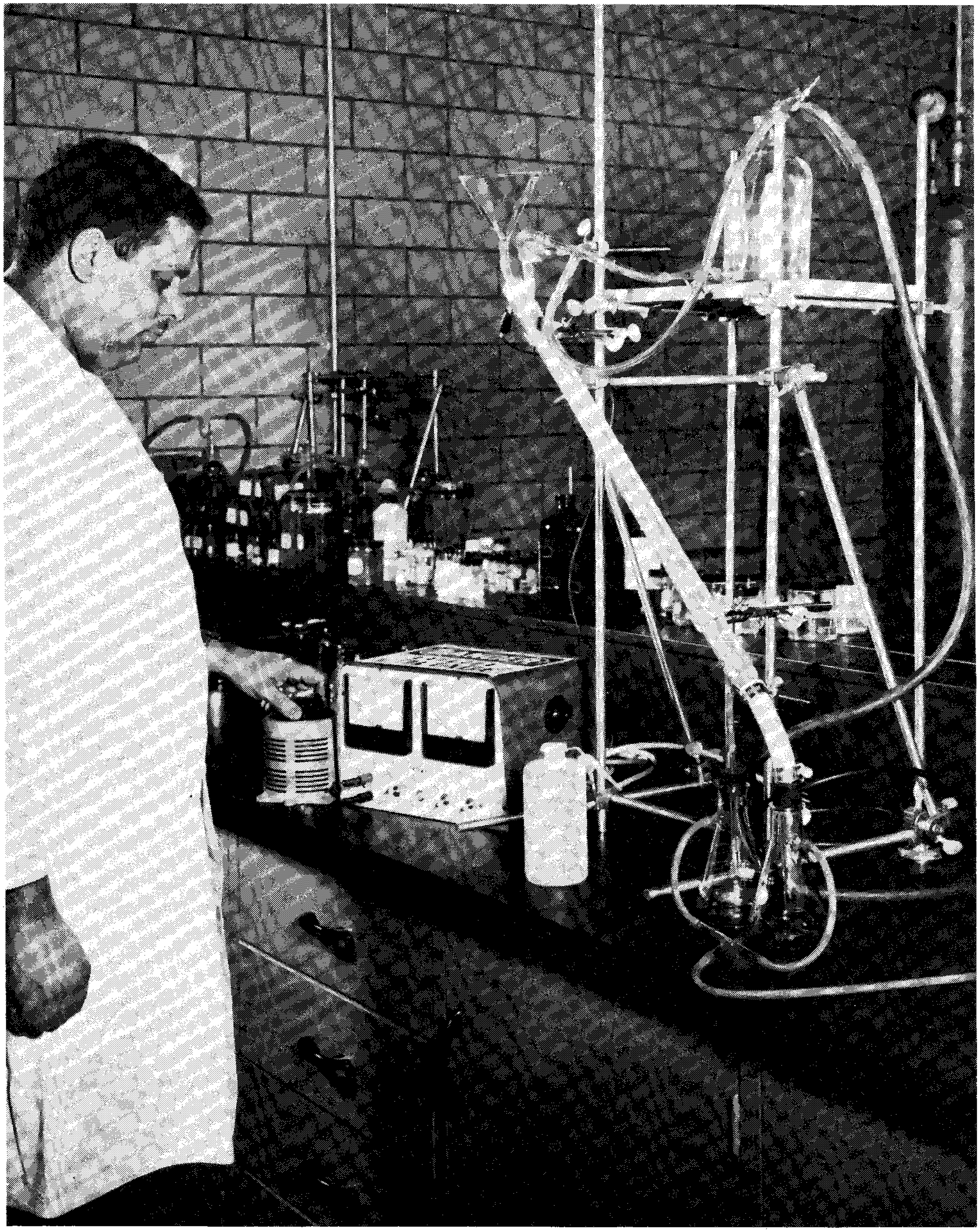

FIGURE 9. - Electrophoresis Column for Pyrite Separation From Coal.

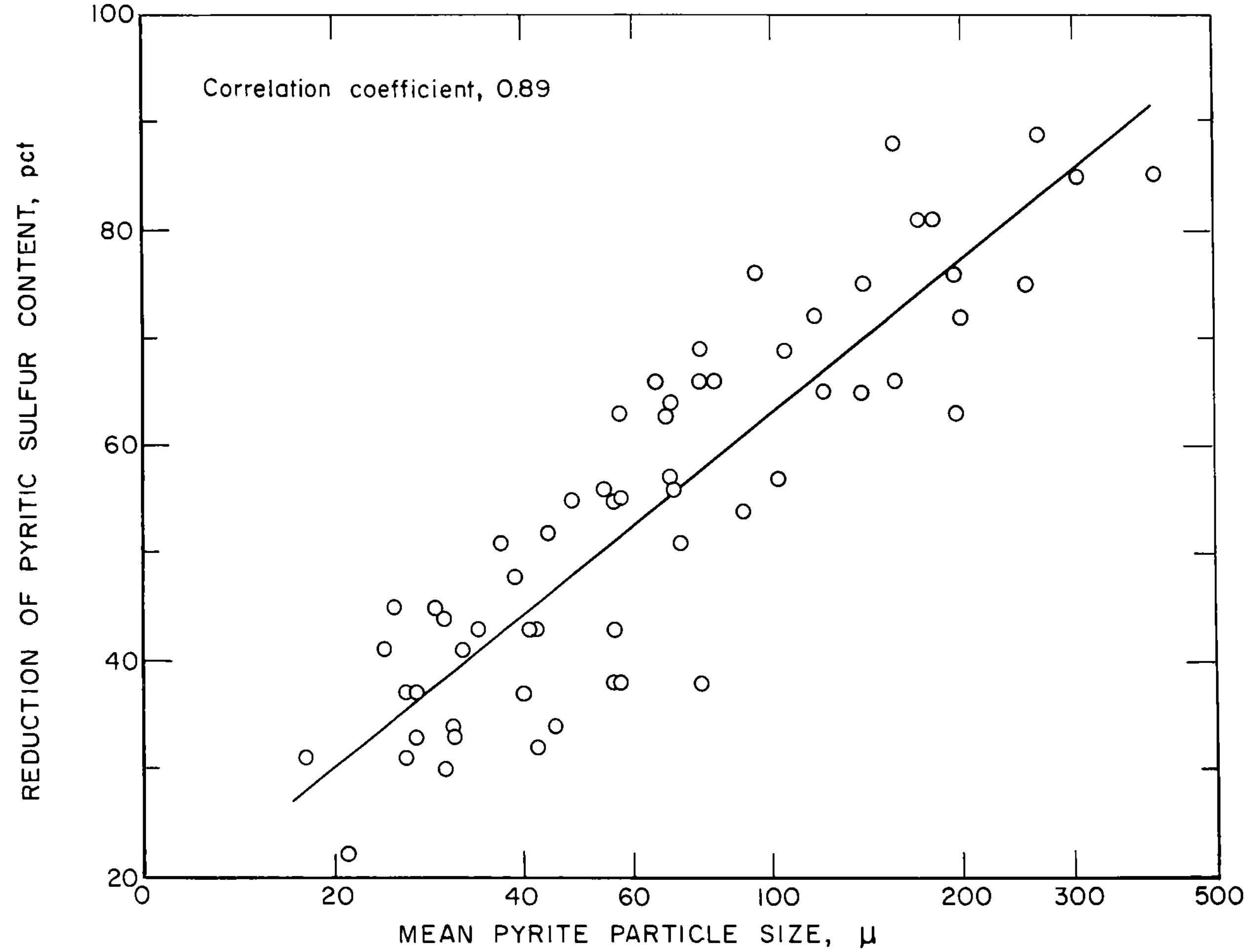

FIGURE 10. - Pyritic Sulfur Content Reduction as a Function of Pyrite Particle Size.

respectively. It is apparent that the extent of pyrite removal can be fairly well predicted from microscopic analysis.

Stack Gas Purification

Sulfur Dioxide Removal From Gases

Alkalized Alumina Process

Alkalized alumina is a highly reactive solid material that will absorb sulfur dioxide from flue gases and can be readily regenerated with recovery of the sulfur in the elemental form. A 500-scfm pilot plant was constructed and operated to remove 90 percent of the sulfur dioxide from flue gas containing 0.2 volume-percent of this air pollutant, without excessive pressure drop. Absorbent losses were found to be excessive, however, making process operation prohibitively costly and preventing long-term operation of the pilot plant necessary to further prove and develop the process.

In early 1969 a program was initiated by the Bureau and others to develop a stronger alkalized alumina. Meanwhile, pilot plant experiments were continued using an inert alumina catalyst support. In continuous operation for 100 cycles, absorbent losses were held to 0.1 percent per cycle or less, and without excessive accumulation of fly ash. An absorbent with acceptable attrition resistance had not been developed by mid-1969, however; pilot-plant operation was stopped. Efforts were continued to produce an acceptable absorbent.

Evaluation of SO_2 Absorbents and Reactants

Many new alkalized aluminalike sorbents, commercial and Bureau-prepared, were evaluated for their regeneration reactions. The chemistry of regeneration of some of these alkalized aluminalike sorbents was quite different than that found for the regular alkalized aluminas. For example, the hydrogen reduction of sodium aluminate on alumina produced sulfur oxides and elemental sulfur, with little formation of hydrogen sulfide. This is compared with the regeneration of the alkalized aluminas, where 80 to 90 percent of the sulfur was removed as hydrogen sulfide. With all the preparations, a small amount of iron was required to make the reduction reaction proceed rapidly, often in less than 1/2 hour. When no iron is present, several hours are required to complete the regeneration.

Another sulfur dioxide sorbent, copper sulfate on alumina, was regenerated at low temperatures (400° to 500° C) using methane, with the sulfurous gas being sulfur dioxide. An important feature of this methane regeneration, besides low temperature requirements, is that approximately 3 moles of sulfur dioxide are produced for each mole of methane reacting.

Promising absorbents developed in bench-scale work were evaluated under realistic conditions. Nonregenerable ores or waste products, such as red mud and trona, were added to the flue gas generated by a laboratory-scale furnace. Sulfur dioxide removal as high as 75 percent was achieved at 600° C with red mud injected at a rate of 23.9 pounds per pound of SO_2. It was indicated that effective sulfur dioxide removal is possible using trona at lower feed rates and temperatures. Testing of regenerable absorbents in actual flue gas was begun.

A number of low-cost ores and waste products, such as red and brown muds (waste products from bauxite refining), oil shales, and nahcolite or trona (natural sodium bicarbonates) were found to absorb large quantities of sulfur dioxide. None, however, were regenerable. Copper salts on alumina have been found to react readily with sulfur dioxide at 300° C (572° F) and to be regenerable with methane at 400° C (752° F). Economic commercial application appears possible, but because the absorbent is relatively expensive, it must last for extended periods, physically and chemically. Lower-cost regenerable absorbents were prepared from various combinations of alumina hydrate, sodium aluminate, sodium hydroxide, red muds, and iron salts; none were strong enough. Efforts were continued to improve their strength and lower the temperature needed for regeneration.

Preliminary studies were completed on the removal of sulfur dioxide from stack gases by reaction with gaseous ammonia.[14] Removal efficiencies of up to 99 percent were obtained by this technique. Bench-scale tests indicate a process cost of slightly more than $1 per ton of coal used. If this cost can be reduced, investigation on a pilot plant scale might be warranted.

Research was conducted on the thermal conversion of pyrite into sulfur and ferrous sulfide. The latter is potentially useful for removing sulfur dioxide from stack gas in a reaction that also produces iron oxide suitable as blast furnace feed. A falling bed reactor was designed and constructed to process pyrite at 1,650° F (900° C) in a nitrogen (inert) atmosphere. Complete conversion of pyrite to sulfur (and ferrous sulfide) was effected successfully with this reactor; the addition of an electrostatic precipitator facilitated the recovery of all sulfur mist from the effluent gas.

Bench-scale studies were continued on the use of reducing gases to effectively regenerate manganese oxide that has been used to absorb sulfur dioxide from flue gas. Efforts so far have been unsuccessful.

Fundamental Research on SO_2 Absorbents

Infrared studies of the sulfur dioxide sorbent, alkalized alumina, showed that simulated flue gas containing this air pollutant and nitrogen oxides partially destroys the sodium aluminate structure. After three cycles of sorption-regeneration the destruction was 75 percent complete. When nitrogen oxides were absent from the gas the aluminate structure remained stable. Also, surfaces of sulfur dioxide sorbents were investigated by the new technique of irradiation with low-energy light from a carbon dioxide laser.[15] The first evidence was obtained for the interaction of nitrogen oxides in flue gas with alkalized alumina sorbent.

Reactions involved in the regeneration of sorbents used for SO_2 removal from flue gases were investigated by continuously monitoring the effluents by mass spectrometry. Evolution rates of SO_2, SO_3, and H_2S were compared for the regeneration of copper sulfate sorbent with hydrogen and methane (fig. 11). Regeneration with hydrogen was the more rapid.

A few differential thermal analyses were conducted on alkalized alumina samples representing various stages of the reaction cycle. Endotherms, as a result of gas evolution, were observed in all samples, and in some, exothermic reactions were indicated. The magnitude of the terms appeared to be a function of the sulfur content or exposure time of the sorbent to flue gas.

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[14] Shale, C. C., D. G. Simpson, and P. S. Lewis. Removal of Sulfur and Nitrogen Oxides From Stack Gases With Ammonia. Paper in Symposium, Important Chemical Reactions in Air Pollution Control, Part II, 62d Annual Meeting, AIChE, Washington, D.C., November 16-20, 1969, 17 pp.

[15] Sharkey, A. G., Jr., A. F. Logar, and F. S. Karn. Laser Irradiation of Oxidized Coal. Fuel, v. 48, 1969, pp. 96-97.

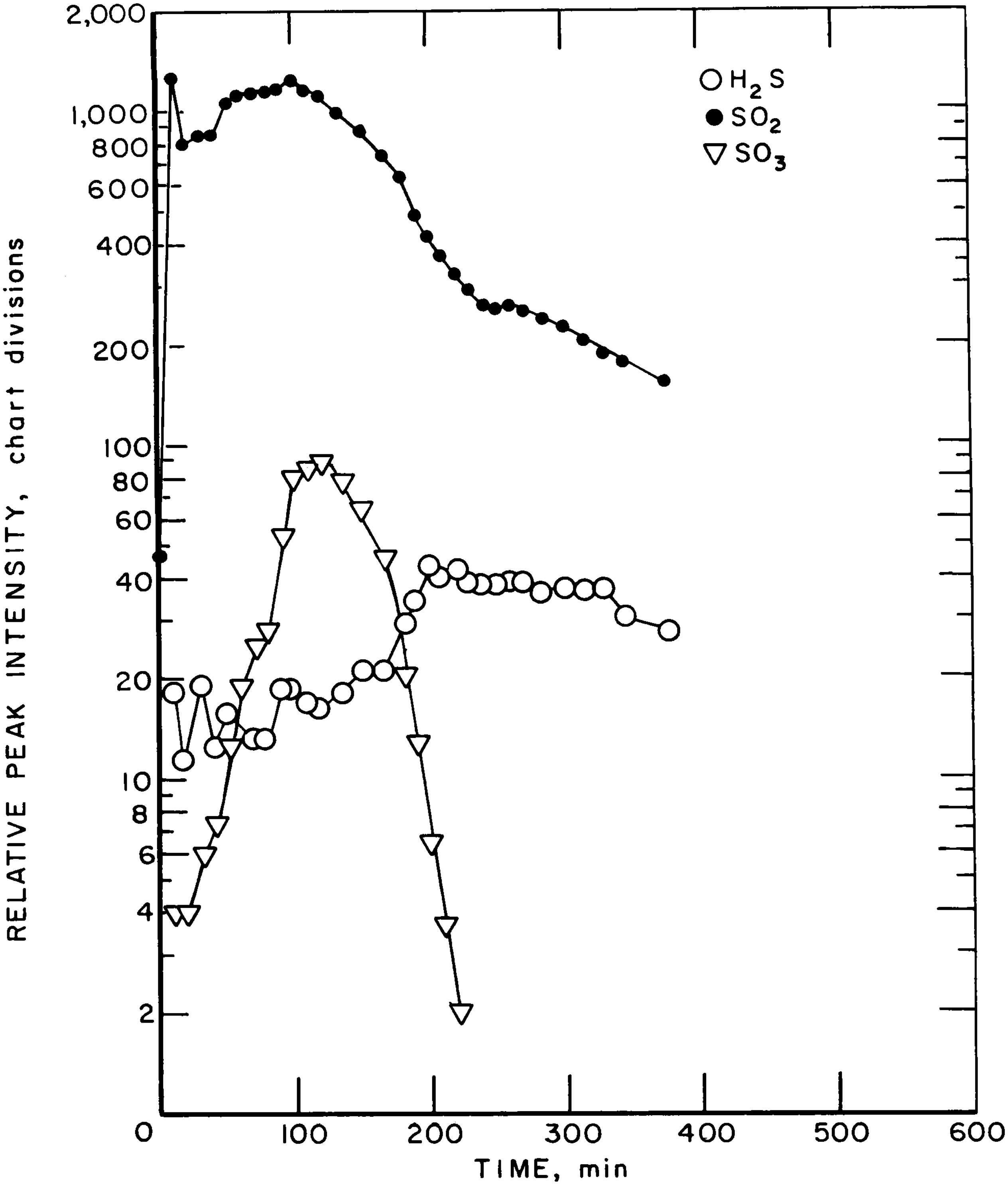

FIGURE 11. - Sulfur Gases From Copper Sulfate Regeneration in Methane.

Instrumentation for the detection of sulfur-33 magnetic resonance (^{33}S NMR) was installed on an existing nuclear magnetic resonance spectrometer.

Signals from ^{33}S are extremely weak and difficult to locate, but a spectrum of carbon disulfide was finally obtained. Resonances have been recorded for thiophene and for carbon disulfide in thiophene; resonances for tetrahydrothiophene and for enriched elemental sulfur could not be detected.

A study has been made on a gas-solid reaction involving sulfur dioxide and an iridium complex imbedded in a potassium bromide pellet. Sulfur dioxide is able to diffuse through the bromide crystallite matrix and react with the complex. By the use of a special cell quantitative infrared spectra were continuously obtained in situ during the reaction. Errors caused by repeated pellet preparation were eliminated. New structures are observable, with the naked eye and spectrally, as they form.

Nitrogen Oxides Removal From Stack Gases

Internal combustion engines and stationary powerplants burning fossil fuels are the major sources of nitric oxide. Cooling of the combustion gas increases the decomposition rate of nitric oxide; however, as the temperature continues to drop, the rate of decomposition decreases rapidly and much of the oxide remains. Nitric oxide decomposition by catalysis is being studied with a bench-scale unit fed with simulated flue gas containing nitrogen, carbon dioxide, oxygen, sulfur dioxide, nitric oxide, and water vapor. Concentrations of the sulfur dioxide and nitric oxide are 2,000 and 500 ppm, respectively; hourly space velocities are 1,000 to 4,000 vol/vol/hr. High initial activity occurs with several catalytic systems, but the degree of decomposition decreases rapidly. In addition to catalytic decomposition, absorption on manganese and nickel oxides followed by a regenerative step is under study.

Chlorine Reduction in Stack Gases

The objective of this project was to determine the nature and quantity of chlorine compounds evolved in the combustion of pulverized bituminous coals, and to develop techniques to reduce the amounts of these effluents. High-, medium-, and low-volatile coals containing 0.08 to 0.39 percent chlorine were burned in a 2-1/2-inch-diameter refractory tube.[16] At carbon-combustion efficiencies of 94 to 98 percent, an average of 4 percent of the chlorine was retained in the ash; the balance, predominately hydrogen chloride, was evolved in the combustion. Sulfur retention in the ash averaged 6.3 percent.

Small quantities of alkali and alkaline earth carbonates added to the coal preferentially reacted with the chlorine. This resulted in an increased chlorine-to-sulfur atomic ratio in the ash. Addition of 2 percent potassium carbonate increased the retention of the chlorine in the ash from 4 to 30 percent and the chlorine-to-sulfur atomic ratio in the ash from 0.22 to 0.75.

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[16] Iapallucci, T. L., R. J. Demski, and D. Bienstock. Chlorine in Coal Combustion. BuMines Rept. of Inv. 7260, 1969, 12 pp.

FIGURE 12. - Developing Analyzer for Size and Concentration of Dust in Stack Gases.

Stack Gas Sampling and Analyzing

Development was continued of an improved method for sampling and analyzing dust-laden stack gases. Numerous techniques are available for sampling and analyzing clean gases, but none are adequate for gases containing dust. A system is being developed to analyze stack effluents, automatically and continuously, for dust size, dust flow rate, and concentrations of constituent gases. An effective system for complete analysis of dust-laden gases would help reduce and control the production of airborne solid pollutants.

In the system being developed, a probe-pitot tube device automatically traverses the stack duct and samples the gas at correct rates maintained by a servocontrol monitor. The sampler and monitor are both undergoing tests on a boiler installation to refine their performance. Being developed for use with the sampler and monitor is a unique analyzer, based on application of electrogasdynamic principles (fig. 12), to continuously and instantaneously determine the size and mass flow rate of dust particles in the sampled stream. Laboratory investigations during 1969 established EGD as valid for the purpose, and work was started on a practical system for field testing.

The complete sampling-analyzing system will also include commercially available instruments to determine the concentrations of gaseous constituents in the stack gases.

Sewage Treatment With Coal

Bench-scale studies were continued of coals and coal-derived materials as adsorbents for contaminants in secondary treated sewage.[17] In addition to removal of chemical oxygen demand (COD), emphasis was maintained on removing nutrients such as phosphates and nitrates, which cause certain types of water

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[17] Johnson, G. E. Process for Removing Organic Contaminants Using Coal Adsorbents. U.S. Pat. 3,424,676, Jan. 28, 1969.

pollution. Coal substances were found to remove little nitrate unless the substance was high in iron content, as is the case with some coal refuse. Coals were not good adsorbents for phosphates, although some coal chars showed an unusually high affinity for them. Certain chars would effectively remove one or possibly two of the contaminants in secondary waste waters, but not all three. Mixing the best adsorbents for each contaminant gave an adsorbent that effectively removed COD, nitrate, and phosphate.

SECTION 2. - COAL MINING

Longwall Mining

In cooperation with Old Ben Coal Corporation, Benton, Ill., surface and underground instrumentation was completed at Old Ben's No. 21 mine longwall operation to develop engineering information relating to safe and efficient designs of longwall mining systems. Surface instrumentation over the longwall panel includes subsidence survey stations, strain rosette arrays, and surface tilt measuring stations.

A 7-7/8-inch-diameter by 610-foot-deep borehole drilled from the surface to within 50 feet of the coalbed being extracted by longwall mining has been instrumented to detect and measure substrata movements. Instrumentation of this uncased, but grouted, borehole consists of a slope indicator casing to permit use of devices to measure magnitude and direction of changes induced along the borehole by mining. Supplementary instrumentation is used to detect shifts in the overburden by means of coaxial cable and hydraulic actuated borehole pressure cells.

Instruments have been installed underground in a selected test area in the longwall panel to determine structural properties, ground stresses, and ground movements. Included are borehole pressure cells, roof and floor convergence recorders, romometer stations, and continuous hydraulic pressure recorders on three adjacent, seven-leg, 595-ton-capacity roof supports. Also, 40-foot and 70-foot boreholes were drilled vertically in the roof for visual exploration and measurement of roof strata changes.[18]

Significant information has been obtained on subsidence over the mined-out longwall panel. Maximum subsidence measured was 3-1/2 inches over a mined-out area approximately 494 by 372 feet. Reportedly, normal subsidence at this mine where pillars are extracted for an equivalent area would be about 2-1/2 to 3 feet. Figures 13-14 show on-site installations of measuring and recording instruments.

Methane Control

Research on control of methane gas in coal mining is divided into two broad areas: applied and fundamental. Applied research efforts are aimed

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[18] Barry, A. J., O. B. Nair, and J. S. Miller. Specifications for Selected Hydraulic-Powered Roof Supports With a Method To Estimate Support Requirements for Longwalls. BuMines Inf. Circ. 8424, 1969, 15 pp.

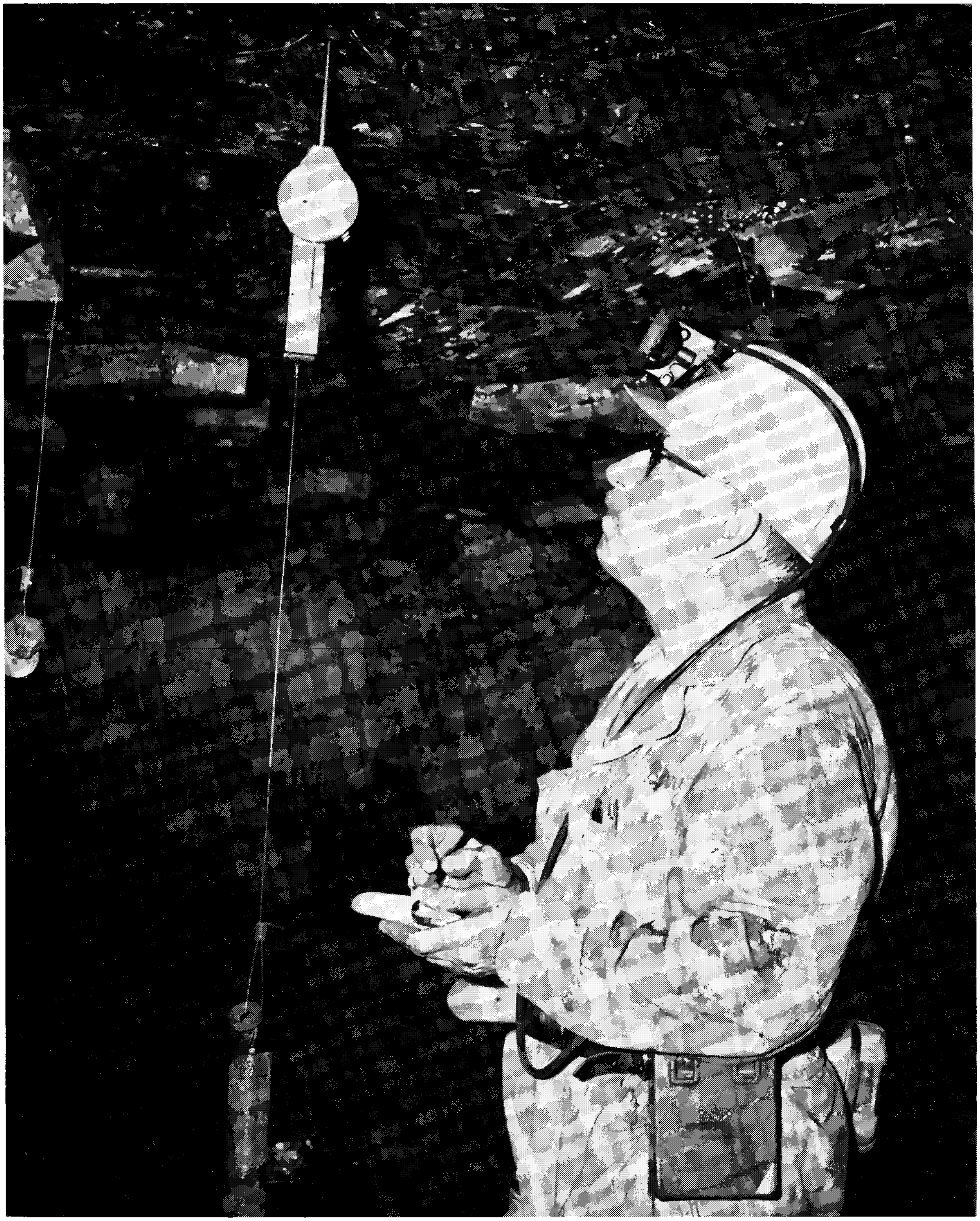

FIGURE 13. - Using 10-Horizon Spring-Anchor Piano-Wire Extensometer To Measure Roof Strata Separations in 70-Foot Borehole Near Longwall Face.

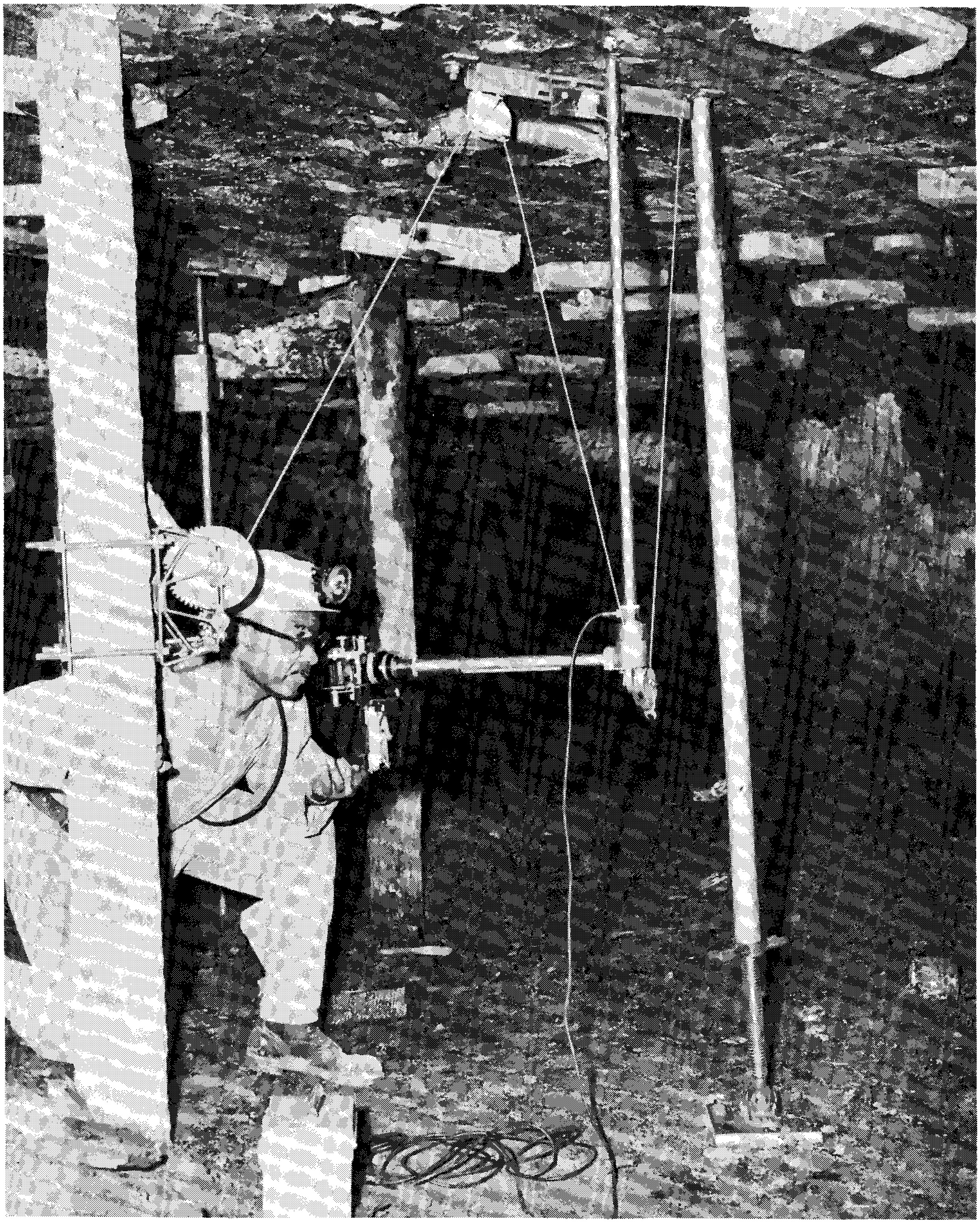

FIGURE 14. - Camera-Borescope Assembly To Record Fractures in Mine Roof Penetrated by 40-Foot Borehole Near Longwall Face.

directly toward improving mining methods and developing techniques for efficiently and safely removing the gas before and during mining. Fundamental research on methane is aimed toward determining the origin, migration, accumulation, and emission of methane from coalbeds and associated strata.

Methane Control in Mining

Methane Drainage Through Vertical Boreholes

Not all of the gas entering mine workings comes from the coalbed. Studies of core drill logs and observations during mining show that gas sometimes exists in the strata above and below the coalbed. In one mine in central Pennsylvania where the overlying strata contained gas, gas entering the mine from gob produced during longwall mining permitted only intermittent operation. An 8-inch-diameter drainage hole to the coal was drilled in advance of the longwall face and cased to 29 feet above the base of the coalbed; the bottom 48 feet of the casing was slotted and left open. The casing above the slotted portion was cemented to the surface. This 77 feet of ungrouted hole provided free drainage of methane from the exposed formation in the borehole and from fractures intercepted by the borehole when the overburden was collapsed. Because of the low permeability of the unfractured formation, however, natural flow from the hole was only 400 cubic feet of gas per day.

A 170-cubic-foot-per-minute explosion-proof vacuum pump was installed on the borehole. Pumping was intermittent with negligible methane flow until the longwall face intersected this drainage hole, and then a sustained flow was established. The pumping rate was adjusted to keep the gas concentration in the exhaust well above the explosion range.

Gas exhausted from the borehole averaged 90 percent methane and 172,000 cubic feet per day of methane was evacuated from the hole during the first month of operation. During a 3-day pump shutdown, the borehole developed a natural flow of 215,000 cubic feet per day of methane, indicating that the pump was actually inhibiting the flow. A 700-cubic-foot-per-minute pump was then installed, and an average of 335,000 cubic feet per day of methane was evacuated during the next 4 months. Averaged methane composition of the gas was 34 percent. Drainage of the gob gas through vertical boreholes substantially reduced the methane content of the ventilation air. Monitoring of the returns off the longwall panel showed a reduction of approximately 1 percent methane in the main air returns ventilating the panel.[19]

The vertical boreholes proved so beneficial that additional holes are being drilled to drain methane from the gob areas of all active longwall panels. A second vertical borehole, with an additional 30 feet of overburden, was exposed to open flow; a more powerful blower extracted an average daily volume of 1 million cubic feet of methane during 2 months of service. The exhaust averaged 88 percent methane.

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[19] Elder, C. H. Use of Vertical Boreholes for Assisting Ventilation of Longwall Gob Areas. BuMines Tech. Prog. Rept. 13, May 1969, 6 pp.

Methane Drainage Through Horizontal Boreholes

Horizontal borehole drilling is being used to evaluate reservoir pressures in coalbeds as well as to obtain coal cores for laboratory studies. Equipment was developed (fig. 15) that can drill a hole 400 feet or more horizontally into Pittsburgh bed bituminous coal at an average rate of 34 feet per

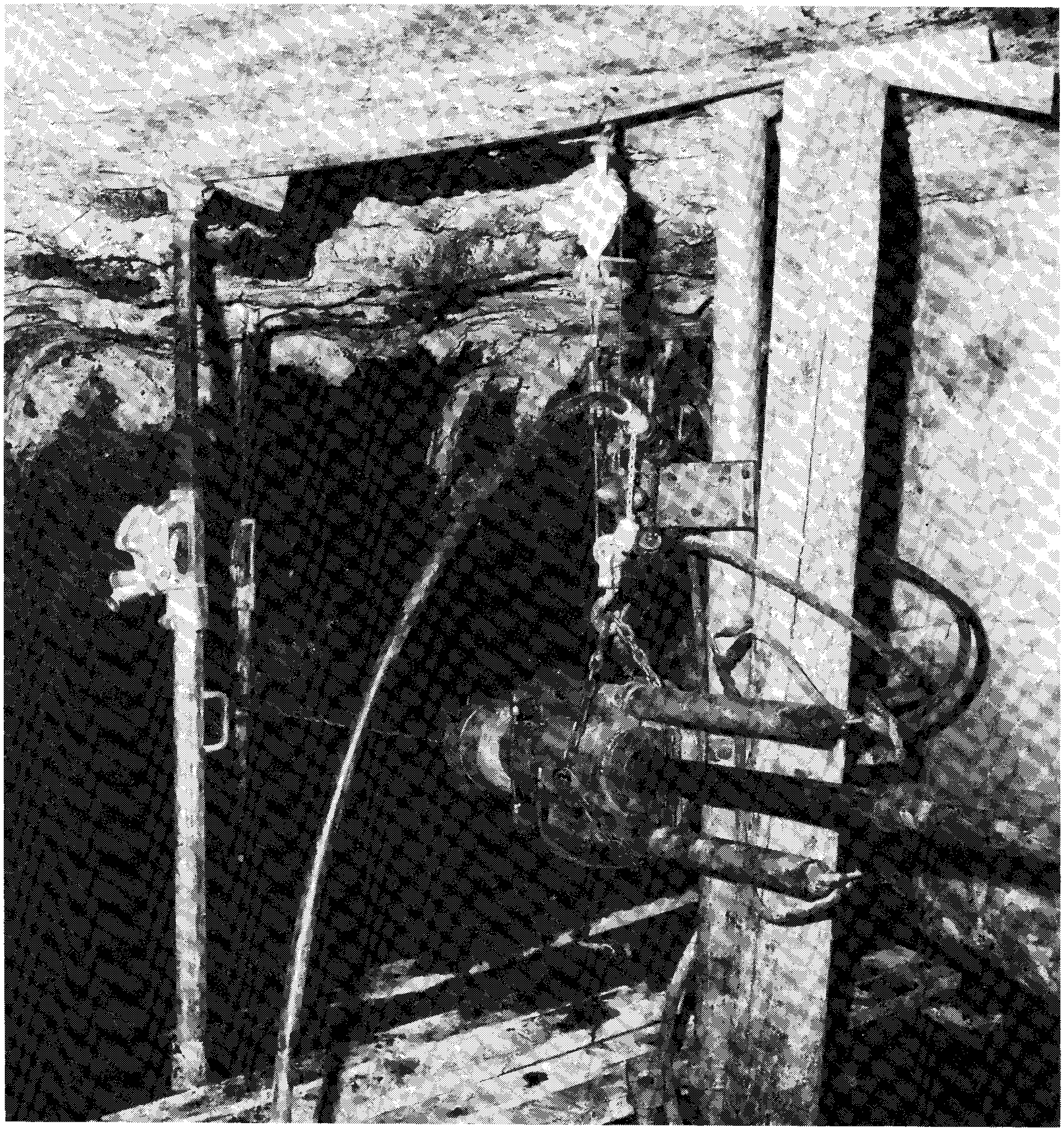

FIGURE 15. - Hydraulic Drill for Horizontal Borehole Drilling.

hour.[20] (Penetration rates of up to 180 feet per hour have been achieved.) Additional research is being conducted to develop methods of drilling horizontal holes without excessive deviation of the bit.

Packers must be inserted to measure pressures at the backs of these holes, and the only suitable packers presently available cost about $500 each and about 20 would be needed to effectively seal a 300-foot drill hole. Alternate designs are being investigated to reduce the cost. Grouting to seal the holes is being investigated, but no satisfactory grouting material or technique has yet been found.

Influence of Mining on Gas Emissions

A study of methane emission rates was conducted in connection with the development of a main headings system in a gassy northern West Virginia mine. The system consists of eight 14-foot-wide by 6-1/2-foot-high headings on 100-foot centers. A full boring-type continuous miner is used to mine the Pittsburgh bed bituminous coal. One side of the section was practically circumscribed with mine workings; the other paralleled virgin coal. The mining machine dropped the coal on the floor, and a conventional unit loaded it into shuttle cars for transport to a belt conveyor.

Intake air was conducted through the middle headings to the working places and returned through the two airways on either side. Total air volume in both splits fluctuated between 58,000 and 66,000 cubic feet per minute during the study period of 5 days. This fluctuation was caused primarily by traveling equipment and line brattice.

Methane emission rates increased generally as mining progressed toward the virgin area. Mining had to be stopped frequently because of excessive methane emission in the heading immediately adjacent to the virgin coal. Correspondingly, the methane volumes emitted were appreciably lower in the left air split than in the right split, 82 to 92 cubic feet and 134 to 169 cubic feet per minute, respectively. Also, production per shift was usually higher on the left split, as was the area of cumulative solid coal surfaces exposed.

Another study of methane emission rates was conducted on a longwall section in the Lower Kittanning coalbed in central Pennsylvania. The longwall face measured 585 by 3,000 feet in length and averaged 54 inches in thickness, with 1,200 feet having been mined out at the start of the study. Mining was conducted with a 15-foot-long bidirectional shearer equipped with a single 48-inch-diameter, double spiral vane drum (adjustable to suit the seam height) and a cowl to move coal onto the face conveyor on the backward run. Roof support was provided by self-advancing (hydraulic) steel prop assemblies.

A total of 81,000 cubic feet per minute of air was used in the system, with 30,000 cubic feet per minute starting at the head end of the face conveyor and 7,000 cubic feet per minute reaching the tail end. Although only

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[20] Hadden, J. D., and Joseph Cervik. Design and Development of Drill Equipment. BuMines Tech. Prog. Rept. 11, May 1969, 11 pp.

0.10 percent methane was detected along the coal face during operation of the shearer, the return airways behind the longwall caved area averaged 1.6 percent. This indicates that practically all of the methane was emitted from caved roof. Previous studies by company and Bureau of Mines personnel in drilling a borehole to the Lower Kittanning coalbed indicated that the methane zone extended vertically about 60 to 70 feet from the bottom of the "rider" seam (20 to 30 feet above the coalbed mined).

Somewhat less methane was liberated during operating periods than during idle periods. This was probably due to delayed caving to the methane emission zone and to the small percentage of operating time during the total study period. Only in the last 5 out of a total of 22 operating shifts did the emission rate increase (from 698 to 827 cubic feet per minute, or 18.5 percent) as mining advanced. Figure 16 summarizes machine operating time, average total volume in the longwall area, and methane emission rates during the 7-1/3-day study period.

Influence of Clay Veins in Methane Release

Drilling studies were continued in Pittsburgh bed bituminous coal in southwestern Pennsylvania to determine the influence of clay veins on the behavior of the gas.[21] Pressure measurements at various distances through a horizontal borehole showed that clay veins isolated certain sections of the coalbed, forming separate cells of coal-bearing gas. Proper drilling techniques can drain the gas from these cells directly into return airways without overtaxing normal mine ventilation. Penetration of these veins during mining can release dangerously high concentrations of methane.

Clay veins, their origin and occurrence, are being studied in an effort to predict and track their course through coalbeds. The term "clay vein" is a misnomer. Actually, they are lithified clay fracture fillings from which sparks can be produced by the action of bits during mining. Since complete penetration of a clay vein can be accompanied by a large flow of methane, chances of an ignition are high. In Pittsburgh bed bituminous coal deposits in southwestern Pennsylvania and portions of West Virginia, the clay seems to have been forced downward into fractures in the coal forming veins. DTA and X-ray diffraction studies show that there is a marked similarity between roof and clay vein material and that only slight similarities between clay vein material and floor rock. Figure 17 shows a clay vein underground in Pittsburgh bed bituminous coal in West Virginia. Field work indicates that clay veins occur most frequently along lines of flexure, such as the axes of synclines and anticlines. The clay probably was not deposited with the coalbed but was pushed in afterwards.

Mine Ventilation Costs

In conjunction with the methane control techniques being studied in selected mines, mine ventilation costs were established for a gassy mine in

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[21] Hadden, J. D., and Albert Sainato. Gas Migration Characteristics of Coalbeds. BuMines Tech. Prog. Rept. 12, May 1969, 10 pp.

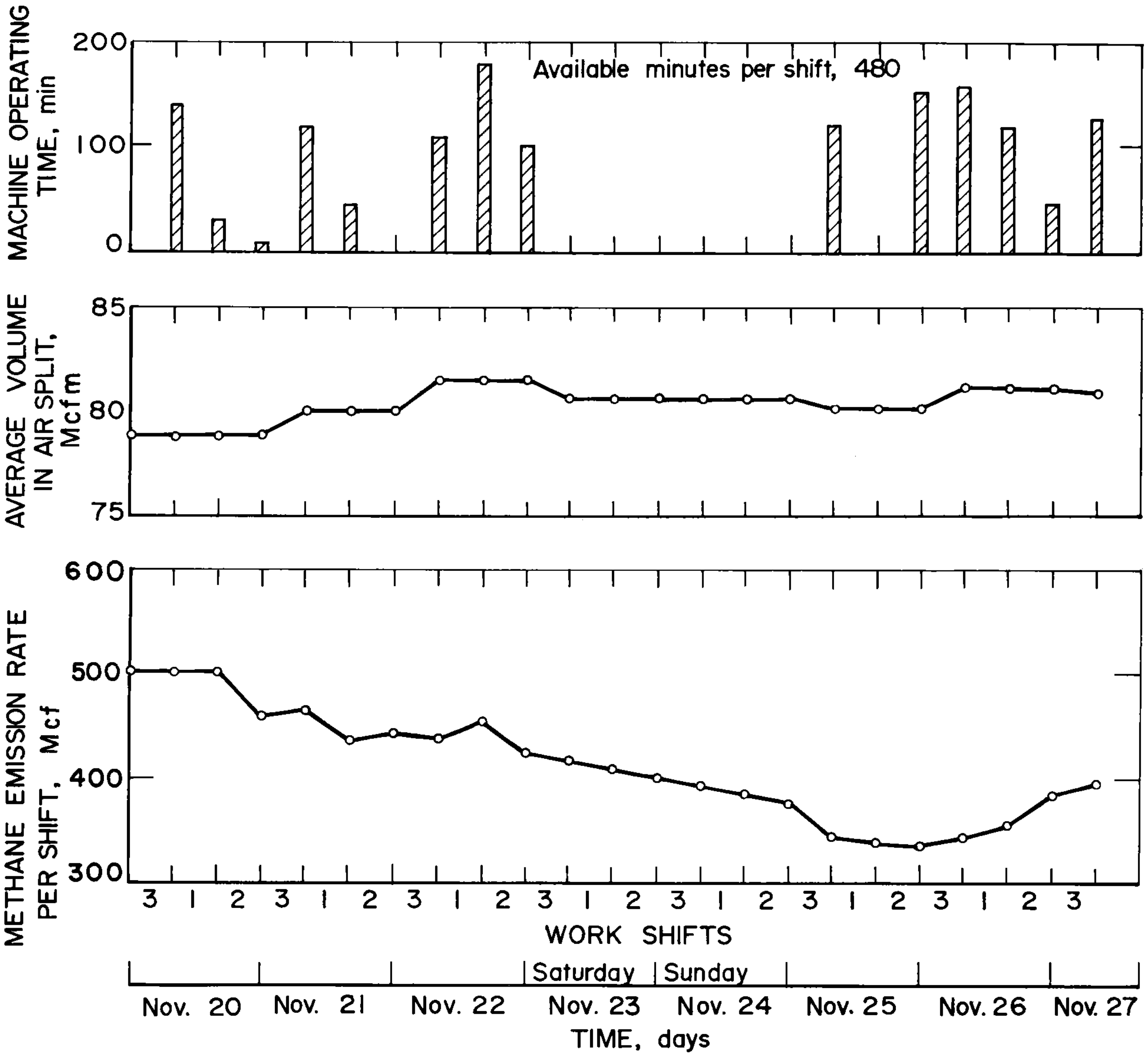

FIGURE 16. - Methane Emission During Longwall Operations.

central Pennsylvania. Included were cost of equipment, openings from the surface for air inlets and outlets, power, and all underground ventilation facilities. Total costs incurred during 4 years 9 months of operation from the date the mine was opened are being reviewed by the company and analyzed by the Bureau to compare (1) the cost of draining methane from five major gob areas through surface boreholes, with (2) the costs of diluting the gas by ventilation.

Air and Methane Flow Meters

Accurate determination of methane concentration in mines is essential to efficient and safe mine management and evaluation of methane-control techniques. Important objectives of the methane control program are the

FIGURE 17. - Clay Vein in Bituminous Coal in West Virginia Mine.

development of standard measurement techniques and a reliable lightweight single instrument package for air and methane flow metering.

Significant improvements have been made in separate instruments for measuring and recording the flow of air and methane in underground coal-mines during the past 5 years.[22] Utilizing these advances, the Bureau encouraged

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[22] LaScola, J. C., and Joseph Cervik. Development of Recording Methanometers and Recording Anemometers for Use in Underground Coal Mines. BuMines Tech. Prog. Rept. 15, May 1969, 17 pp.

development of increasingly sensitive and reliable instruments and stimulated industry to develop a combined instrument. Specifications were drawn incorporating such features as miniaturization, dual track recorder, lightness of weight, ease of operation, internal calibration, and reliability. Design proposals for construction of a combined anemometer-methanometer instrument were requested from leading instrument manufacturers. Subsequently, several proposals were received utilizing different sensing techniques. Evaluation of these proposals is in progress, and meetings have been held with company officials to discuss and finalize specifications. A two-step formal advertising procedure is being followed for the design, development, and construction of this instrument. To establish the performance characteristics of these instruments, plans have been made to test them in the laboratory and under field conditions.

A wind tunnel was designed and fabricated for the calibration of anemometers, and a hot-wire anemometer was purchased capable of sensing airflows as low as 3 feet per minute. Also acquired was a specially designed wind tunnel calibrator (fig. 18) to accurately establish hot-wire anemometer performance that will serve as a standard for air velocity measurements. Hot-wire calibration tests have been completed, and velocity profiles and intensities have been determined in a large wind tunnel (fig. 19). Field-type Biram anemometers and methanometers are now routinely calibrated using the wind tunnel.

Fundamental Methane Research

Methane Formation

The fundamental processes that produce the methane found in coalbeds are being investigated as a possible guide to devising control measures. Experiments were begun to determine the gaseous decomposition products of wood shavings, cellulose, and a lignin derivative. These experiments were started after the products had been inoculated with a soil suspension to facilitate microbial reactions such as might have occurred when coal was formed millions of years ago. So far, hydrogen, methane, and small amounts of carbon dioxide, ethane, and ethylene have been detected. Microbial reactions proceed very slowly, however, and sufficient time has not elapsed to determine if differences in gas composition can be related to differences in the original sample material.

Methane Release and Migration

An instrument has been developed that can measure the desorption rate of methane from coal produced underground by various types of mining machines. By means of this device, measurements relating to release of gas from solid coal underground can now be made in the laboratory. Values attained with the instrument agree with measurements by other researchers using different methods,[23] and the instrument is more sensitive and versatile than other instruments built in this country or in Europe. Information obtained by a number of

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[23] Perkins, J. H., and Joseph Cervik. Sorptive Investigations of Methane on Coal. BuMines Tech. Prog. Rept. 14, May 1969, 6 pp.

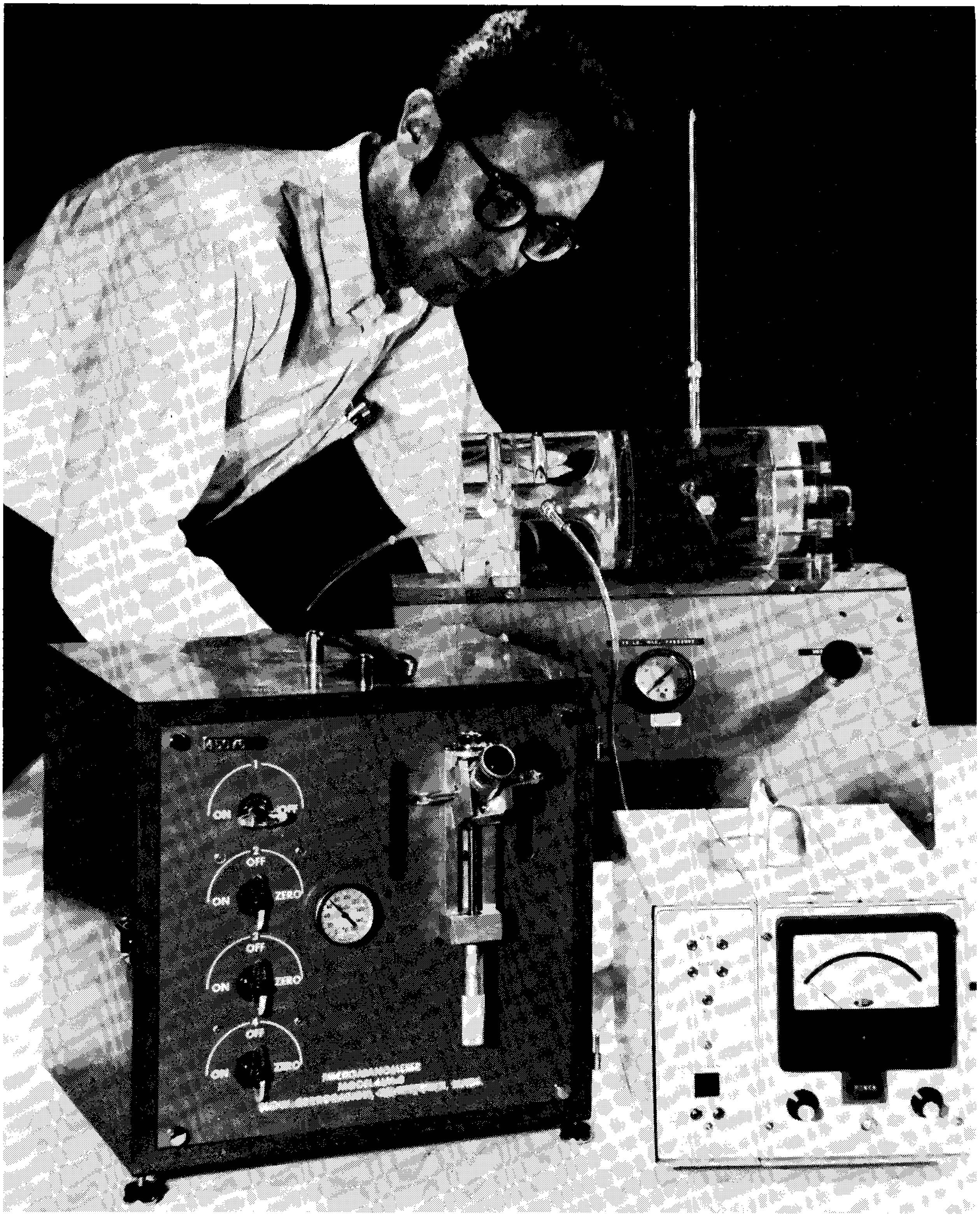

FIGURE 18. - Anemometer Calibrating Unit.

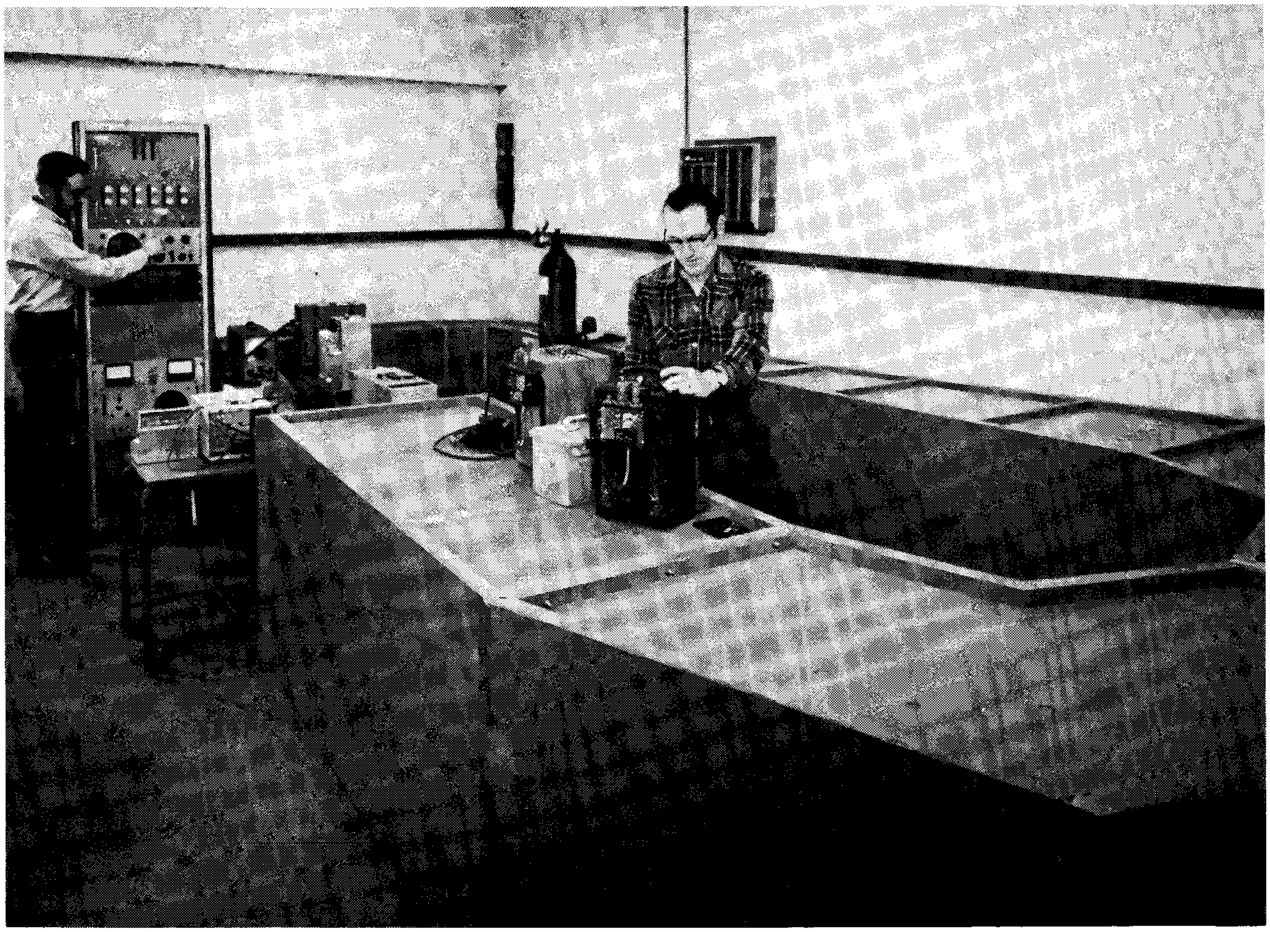

FIGURE 19. - Wind Tunnel for Calibrating Anemometers for Field Use.

such instruments may prove useful in predicting the flow rate of methane from coalbeds into mine workings. Except for fracture porosity, all essential physical properties relating to the transport of methane through a coalbed can be determined.

The volume of gas that migrates into mine workings through fractures in the coalbed is far greater than that which diffuses through the solid coal. The origin and distribution of these fractures is therefore the subject of intensive study. To aid in this, a computer program was developed that graphically depicts the distribution in orientation of fractures observed within an area. A diagram that was developed from 135 data points shows that two sets of fractures exist throughout the area in which the data were gathered--one bearing N 65° W dipping 90°, and another N 29° E dipping 88° NW. Manual plotting of steriographic diagrams is tedious and prone to human error. Computer use eliminates these drawbacks.

A new technique to determine gases released during the fracturing of coal could have an important bearing on mine safety. Newly mined samples of coal are crushed under liquid nitrogen and the condensible gases analyzed by mass

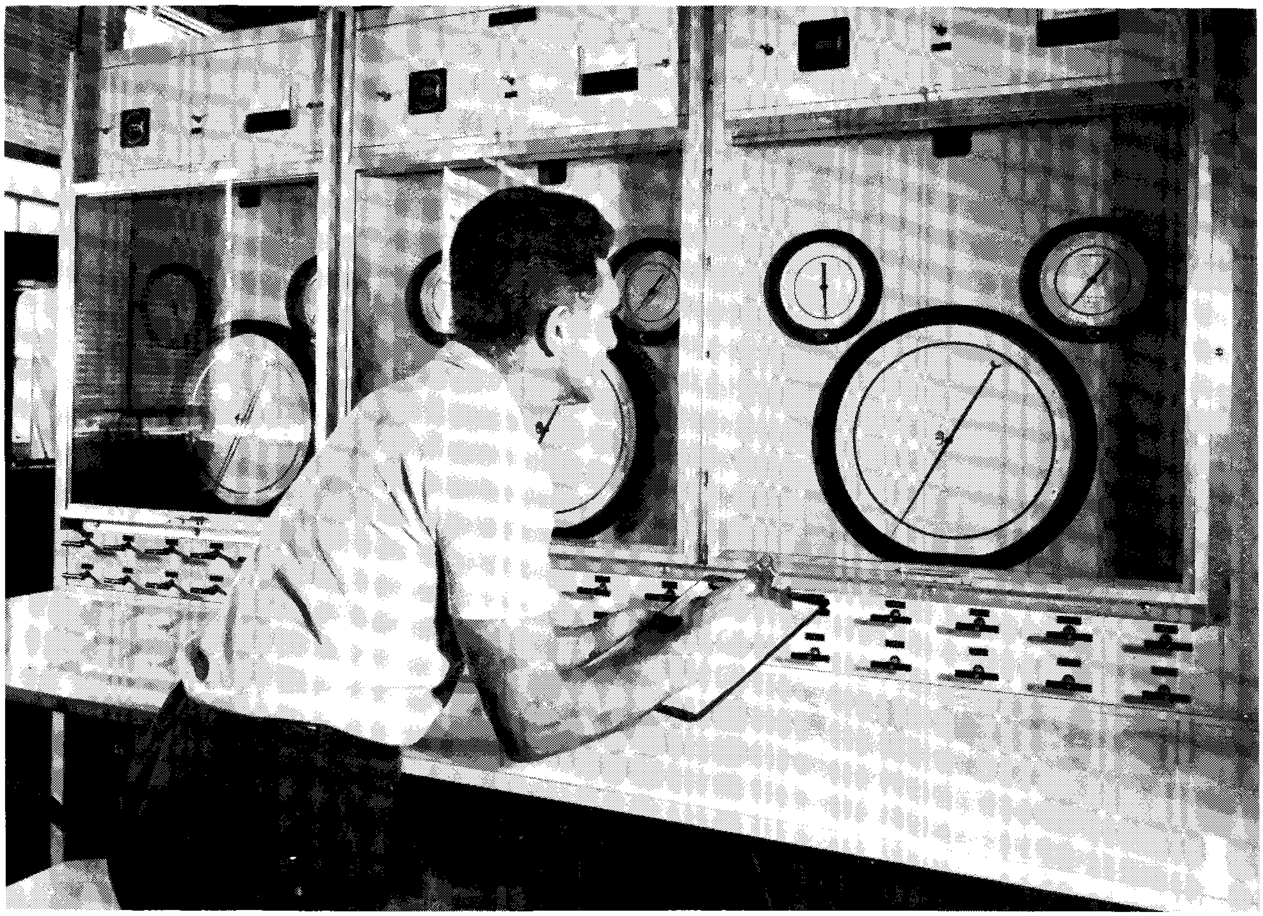

FIGURE 20. - Determining Methane Adsorption on Coal.

spectrometry. Composition of gases released from six different coals were found to vary. In other work, measurements were made of the diffusion rate of methane, ethane, and butane along the bedding plane of Pittsburgh seam bituminous coal.[24] Methane was found to diffuse through coal faster than ethane and butane.

Experiments were conducted to determine the amount of methane adsorbed at equilibrium as a function of pressure on a high-volatile Pittsburgh seam bituminous coal. Basic information was obtained on the subject, and apparatus was constructed for further adsorption measurements (fig. 20).

SECTION 3. - HEALTH AND SAFETY

A mine explosion and subsequent fires in a mine near Farmington, W. Va., in November 1968 claimed the lives of 78 men and aroused public indignation

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[24] Karns, F. S., A. G. Sharkey, Jr., B. M. Thames, and R. A. Friedel. Gas Transport Through Sections of Solid Coal. Preprints, Div. Fuel Chem., Am. Chem. Soc., v. 13, No. 1, April 1969, pp. 130-133.

for stronger mine health and safety legislation. Immediately thereafter, both the Senate and the House of Representatives began considering numerous proposals submitted by various groups interested in mine safety. On December 30, 1969, President Nixon signed a new mine health and safety law designated as the Federal Coal Mine Health and Safety Act of 1969. This act contains the strongest provisions ever provided to protect the health and lives of coal miners. Some of the more important provisions of the new law require improved ventilation practices, removal of the former nongassy designation of certain mines, permissible face electrical equipment, and a specific roof support plan for the face areas of each underground mine. The new law also increased the minimum number of yearly Federal inspections from one to four and requires spot or partial inspections of the more hazardous mines at least every 5 working days.

Provisions never before appearing in coal-mine legislation includes a maximum allowable concentration of airborne dust in working areas, sanitary toilet facilities and potable water at convenient locations underground, penalties for violations of any provision of the act, and a provision that mandatory standards must be promulgated for strip and auger mines.

Coal-Mine Inspection

Coal-mine inspection is designed primarily to conserve human resources by means of inspections, investigations, safety education, and research. Before December 31, 1969, the principal authority for the program was the Federal Coal Mine Safety Act, which, as amended, included certain mandatory provisions applicable to all underground coal mines. Approximately 3,800 underground and 1,700 strip and auger coal mines were in operation during 1968 in 28 States. About 9,100 inspections were made during 1969. Since the first Federal legislation dealing exclusively with coal mine safety was enacted in 1941, more than 228,000 regular Federal inspections of coal mines have been made throughout the industry, along with numerous special inspections, investigations of serious and fatal accidents, and engineering studies. At the beginning of 1969, 251 Federal coal mine inspectors and 14 engineers assigned to coal mines were employed in the five Coal Mine Safety Districts of the Bureau.

Laboratories are maintained at Pittsburgh, Pa., Mt. Hope, W. Va., and Denver, Colo., primarily for the analysis of air and gas samples collected by Federal Mine Inspectors to substantiate their conclusions on the quality of air in mines during regular inspections or to provide scientific guidance when a fire occurs that necessitates sealing a mine.

Coal-mine injury statistics before and after 1941 testify as to the effectiveness of the inspection program. Substantial reduction has taken place in the number of major disasters and fatalities and in the number and frequency of injuries, as shown in the following:

Coal-Mine Injuries

Period[1]	Number of injuries		Injury rate[2]		Percent change			
			Fatal	Nonfatal	Number		Rate	
	Fatal	Nonfatal			Fatal	Nonfatal	Fatal	Nonfatal
1932-41...	12,311	598,438	1.50	72.8	-	-	-	-
1942-51...	10,426	526,880	1.18	59.4	-15	-12	-21	-18
1952-61...	4,021	178,965	1.03	45.8	-61	-66	-13	-23
1962-68[3]..	1,840	74,306	1.07	43.1	([4])	([4])	+4	-6

[1] Calendar years.
[2] Per million man-hours of exposure.
[3] Subject to revision.
[4] Not a comparable period.

Major Coal-Mine Disasters

Period[1]	Major disasters	Fatalities	Fatality rate[2]	Percent change		
				Number of--		Fatality rate
				Disasters	Fatalities	
1932-41.....	45	808	0.10	-	-	-
1942-51.....	43	880	.10	-4	+9	0
1952-61.....	16	199	.05	-63	-77	-50
1962-68[3]....	11	199	.12	([4])	([4])	+140
1969........	0	-	-	-	-	-

[1] Calendar years.
[2] Per million man-hours of exposure.
[3] Subject to revision.
[4] Not a comparable period.

Mineral Industry Health

A section responsible for health conditions in the mineral extractive industries was established in February 1969. The section contends with all health problems such as dust, sanitation, and noise. The section is being staffed to assist compliance with the provisions of the Federal Coal Mine Health and Safety Act of 1969. While helping to collect information to support and justify the provisions in the new health legislation, the group has also conducted studies necessary to evaluate existing mine conditions and set standards for health conditions in mines.

Field organizations have been established at Pittsburgh, Pa., where laboratory facilities are available, and at Denver, Colo., where work was begun on a new laboratory. Plans are underway to establish an automatic weighing system with computer and automatic data processing inputs to weigh the numerous samples of respirable dust to be received by the Bureau for analysis.

The health group presented a symposium on respirable coal mine dust attended by more than 300 persons representing the coal industry, universities, equipment manufacturers, and other Federal and State agencies. Proceedings of this symposium will serve as a "textbook" on respirable dust control for the mining industry. Several members of the group visited Great Britain and

European countries to determine how foreign practices might be utilized in U.S. coal mines. The group also prepared papers for meetings and collected much valuable material on mine health that may eventually be published as a guide.

Accident Prevention and Safety Training

During 1969, an Office of Education and Behavioral Science was established within the Health and Safety Activity to coordinate all the safety education and training activities made available to the mineral industries by the Bureau.

The major goals of this office--safe and healthful conditions in the mineral extractive industries--will be sought through intensive health and safety promotional programs, field investigations, research, accident analysis, and the development of improved teaching methods and techniques.

Consideration will be given to safety and health factors such as dust, noise, and heat to delineate the corrective measures that may be applied and to design training programs adapted to this end. The production of movies for safety training are also an important part of the work.

During fiscal year 1969, 255 classes in accident prevention were conducted by 60 Bureau instructors. A total of 10,105 persons received accident prevention training, of whom 4,912 were in the coal industry, including 1,048 supervisory employees. Another 1,494 persons attended the course, "Use and Care of Permissible Mine Safety Lamps." Coal mining personnel trained in 1968 totaled 4,435.

Each of the Bureau's nontechnical training courses has been revised so that it may be taught in all of the mineral extractive and allied industries. Except for First-Aid, the training course "Principles of Accident Prevention," taught in both the coal and noncoal industries, again elicited the greatest response from industry--1,009 persons in the coal industry and 2,407 persons in noncoal industries.

At outlying small coal mines lacking suitable classroom facilities, training continued to be carried out in mobile units during the fourth year they have been in service. Training in the mobile units is summarized as follows:

	1968	1969
Mines served	80	74
Total employment at mines served	1,889	997
Average employment per mine	24	14
Number of persons trained	1,458	772
Percentage of total employment trained	77	77
Percentage of total accident-prevention training conducted in mobile units (coal industry)	33	12

Men trained in 1968 and 1969 in the coal industry numbered as follows:

	1968	1969
Accident prevention	4,435	4,912
First aid	6,741	5,418
Mine rescue	487	352

Safety education in West Virginia continued to be implemented by participation in the West Virginia Mine Safety Association, a statewide affiliation of the coal-mining industry, mine workers, West Virginia Department of Mines, and the U.S. Bureau of Mines. The association promotes foreman-led weekly safety meetings at coal mines, featuring exchanges of information on accidents and other safety information prepared for the association by the Bureau of Mines. Such information is also distributed to out-of-state coal mining companies, publishers, the Bituminous Coal Operators Association, the United Mine Workers of America, and each Bureau of Mines Health and Safety field office.

In 1968, the Safety Education and Training Group experimented with a series of new training courses in Job Safety Analysis. Twelve coal mining occupations which account for 98 percent of the fatalities in the industry were studied. These courses were well-received in pilot training programs during 1969. The Motion Picture Production Unit completed four films on supervisory responsibility in the metal-nonmetal industries, and is preparing to release films on "Fight Underground Fire," "The Air You Breathe in Industrial Environments," and "Protection Against Radiation in Uranium Mines," each of which will be used in accident-prevention training classes. Bureau of Mines personnel devoted considerable attention to the organization of 46 new chapters of the Holmes Safety Association during fiscal year 1969. It is clearly shown in the series of annual reports of the association that the combined average fatality rates of the affiliated coal mines continue to be much lower than those of nonaffiliated mines.

Roof-Control Research

A new type of roof truss involving a different theory of roof control has been installed in several mines, with good success. Information from these installations is being used to help establish proper installation procedures and the probable results of this type of roof support.

Studies on the stability of mine roof obtained by comparing theoretical data against actual observation have provided a basis from which new programs for roof control can be formulated, resulting in improved safety conditions underground.

An 18-month study was completed on the effect of bearing plates on the performance of rock bolts. Factors examined included plate shape, size, and hole diameters; rock type, characteristics, and surface smoothness; wood headers; bolt length; installed load; ambient temperature; and relative humidity. Tension loss ("bleed off") at the bearing plate end of a bolt was affected by the initial load and by the characteristics of the surface (rock, wood, or steel) against which the plate bears. Relative humidity was critical when shale rock was involved. Plate parameters were not significant; however, when the load on a plate was increased, as happens when the roof moves, the plate shape and size had a significant effect on the integrity of the plate-to-rock interface and the ability of the bolt to restrict movement. It was concluded that bell-shaped plates, 6 by 6 inches or larger, should be used against relatively flat roof surfaces when plate deformation is encountered, and when the humidity fluctuates during the year. Wood headers should not be used except with extremely brittle roof rock where "bleed off" and deformation must be minimized.

Radiography was used to examine the anchorage of bolts in coal, shale, and sandstone. Anchorage is the key to roof bolting, and this new technique may help in selecting the best anchor. The reaction of the rock to the bolt is recorded on film when gamma rays penetrate a 10-inch-diameter core. The radiographs make it possible to observe progressive fracturing, borehole deformation, contact between anchor and rock, and induced strains.

Positive methods are being developed to permit miners and inspectors to judge the adequacy of a roof-bolt plan. One promising technique involved pulling from four points in the roof and measuring the movement of the central span as a function of time and level of applied load. Evaluation of the standard roof bolt anchorage test showed it to be independent of rock creep and tension "bleed off;" results of the test depend principally on the rock in which the bolt is anchored.

Reproducible and reliable mine modeling data were obtained by encasing strain-gage networks in epoxy (fig. 21). Solutions obtained with these models were in agreement with applicable theories and with solutions obtained by the more difficult and complex techniques, such as photoelasticity and finite elements.

A systematic analysis of case histories of falls of bolted mine roofs based on mining company records, field studies, and Bureau reports indicate that much remains to be done to improve roof control safety. A technical basis for the design of roof-control plans is urgently needed. Statistics show that a disproportionate number of injuries occur in small mines, particularly those operating in seams thinner than 60 inches. The incidence of accidents in conventional, hand, and auger mining also appears high.

A project was initiated to study the overall competency of coal mine roof rocks by ultrasonic techniques. Seismic means have been used with encouraging results by previous Bureau investigators to detect loose or "drummy" roof rocks. Three seismic techniques were investigated in the laboratory:
(1) Application of continuous waves, with the wave frequency varied to make loose segments of rock vibrate at natural resonant frequencies detectable as an increase in rock vibration amplitude; (2) detection of packets of sine waves reflected from a discontinuity to determine their travel time in the rock--a measure of the distance to the fracture or parting; (3) application of square pulses of power resonant transducers for use in the same manner as the packets of sine waves. Procedures (1) and (2) were found to impart more energy to the rock and the required voltages were quite low, resulting in

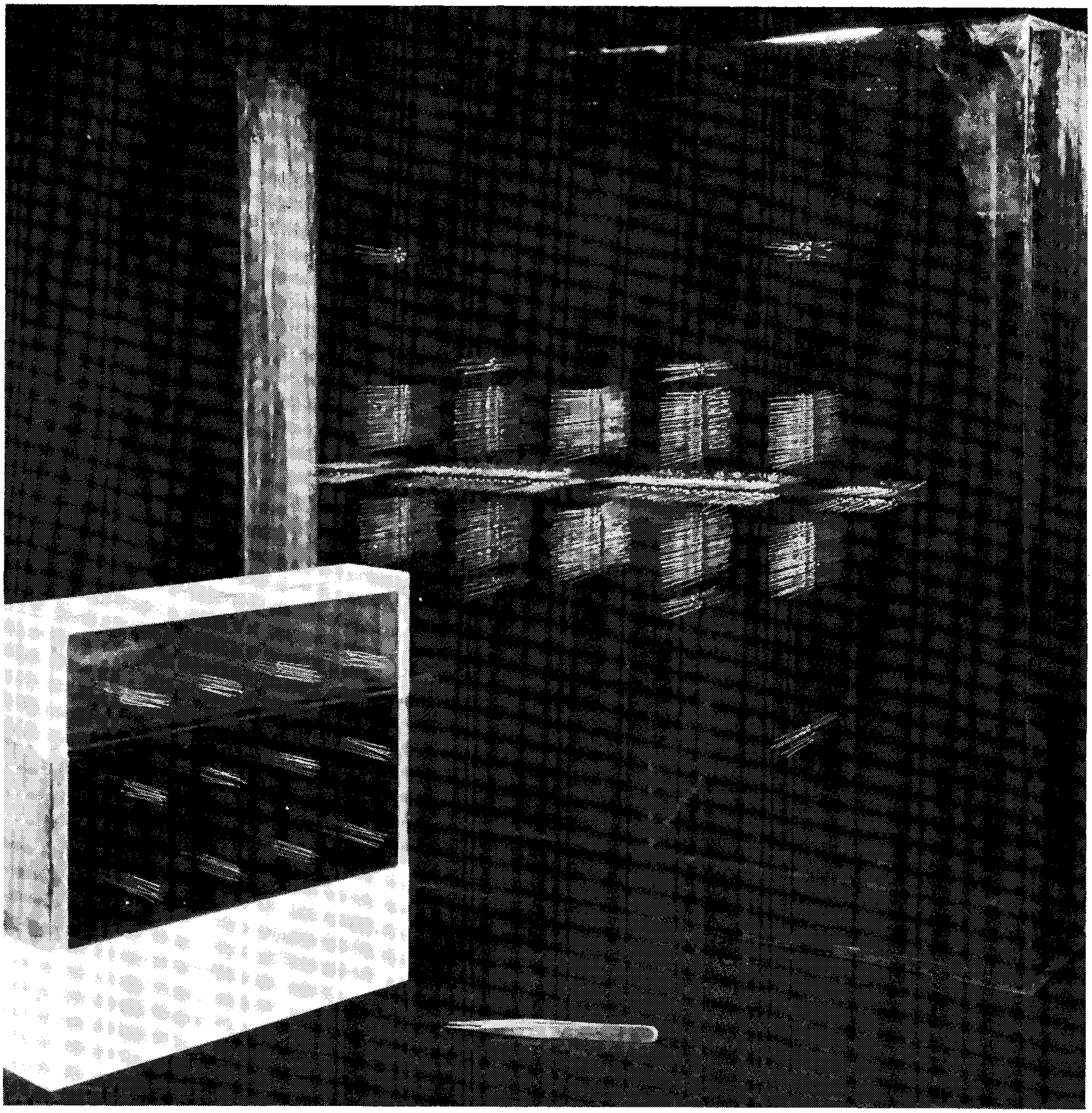

FIGURE 21. - Epoxy-Encased Strain Gage Networks.

greater safety and portability. High-sensitivity resonant transducers of the piezoelectric type have been developed and will be used in conjunction with a tone burst generator (to generate packets of sine waves), an oscilloscope, and an amplifier. Forthcoming field tests of the instrumentation at a dimension stone quarry in which a known fracture has been induced in a semi-infinite rock mass should permit comparison and selection of the best seismic technique for use in underground coal mines.

A study was initiated to explore the possibility of applying statistical correlation techniques to the prediction of bad roof conditions in coal mines.

Mine Air Research

Methane and Other Gases

Two standard analytical procedures were developed. The first is used to determine hydrogen and helium in methane-rich borehole samples taken from coalbeds and from mine-fire seals. The high level of methane in these samples requires considerable variation from the routine mine-air analysis. The second method makes it possible to carry out a large number of gas chromatographic analyses of mine air without disassembly or column change.

Test equipment was developed to evaluate the efficiency of various commercial gas detector tubes. Test results were used as a basis for recommendations to mine inspection personnel as to the best available detector brands for specific applications.

Additives which have been recently introduced for use as ignition accelerators in diesel fuel have raised the possibility of a significant increase in concentrations of nitrogen oxides and carbon monoxide in diesel exhaust. A study, using amyl nitrate and nitrite as additives, revealed no significant increase in the relative amounts of these pollutants in the exhaust gases.

Coal Mine Dust

Measurement of environmental dust concentrations in operating underground bituminous coal mines was extended to 29 large and 12 small mines. The resulting information has been applied in establishing operating standards to protect bituminous coal miners from pneumoconiosis.

Research efforts were intensified to eliminate health hazards created by respirable mine dusts. Theory and experiment are being combined to study the minute (less than 5-micron) particles of coal, rock, pyrite, and other dusts responsible for pneumoconiosis. The resulting information will be applied to developing practical procedures to reduce these dusts to physiologically tolerable levels in underground mines. Initial efforts have been directed toward particle diagnostic techniques and the dust-allaying potential of water spray, steam, and borehole stemming.

Study of the fragmentation characteristics and fragment-size distribution of coal was begun for determining the influence of mining machine operating characteristics on the production of particles below 10 microns. Particle size measurement and other particle characterization in this range depend largely on microscope techniques. A simple technique was developed for examining small coal dust particles with a scanning electron microscope. This technique, which promises to be useful in dust studies, will be discussed in a forthcoming article.

Analysis of total silica in respirable coal dust samples of 5 milligrams or less becomes increasingly important with the advent of new mining legislation. However, the silica content of such samples can be too low (less than 50 micrograms) to permit using standard procedures of analysis. Modifications of several methods were applied experimentally to the analysis of total silica concentrations. Reduction of molybdic silicic acid appears to be most suitable for the purpose.

A standard method was developed for determining low levels of iron in respirable coal dust. The method involves atomic absorption using sodium tetraborate fusion. Spark-source mass spectrometry is being investigated for analyzing trace elements in the parts-per-million and parts-per-billion range that could present health hazards in respiratory-size coal dust.

Sodium chloride aerosol, generated ultrasonically and treated to the desired relative humidity, particle size, and concentration, was explored as a standard aerosol for testing dust filters. It is considered fairly representative of particles that may be encountered in mining and industrial practice.

An innocuous halocarbon was investigated as a substitute for highly toxic carbon tetrachloride used in the approval testing of chemical cartridges. The vapor of 1,1,2-trichloro-1,2,2-trifluoroethane (Freon 113) is continuously monitored by a nondispersive infrared analyzer. Breakthrough times for the cartridges are shorter with the halocarbon, but can be equated to those with carbon tetrachloride by using an appropriate factor.

A standard method was developed for the rapid determination of absorbed water in respirator cartridge material and in explosives.

Work was continued on developing a portable meter to check the effectiveness of rock-dusting procedures. A device utilizing low-energy gamma rays was shown to be feasible for this purpose, and a prototype meter for test purposes is being fabricated. Several instrument companies have expressed interest in manufacturing mine dust meters for industrial application. Development is also underway on a meter to scan the top 1/8-inch layer of mine dust and give readings on the combustible content. The proposed method is based on backscatter of beta rays from deposits of dust from which representative samples are not easily obtained.

Mine Ventilation

Mine ventilation is playing an important role in achieving safer mine operation and improved working conditions for the miners.

Studies were conducted to measure the effectiveness of bleeder systems to control the movement of methane in "gob areas" at 11 different mines. These studies proved that a properly designed bleeder system, effectively maintained, will satisfactorily control the movement of these "gob" gases. In separate studies, the use of underground secondary air circuits (auxiliary ventilation) using auxiliary blowing fans on face areas was found to effectively dilute and remove methane, reducing the hazards of operation. Also, studies at seven different operations determined that secondary air circuits using properly designed and installed auxiliary fans will reduce the amount of dust to which the workers are exposed in face operations.

Mine Equipment and Materials

Permissibility approvals were given for 77 electrically powered mine machine assemblies and accessories; certifications were granted for 41 components. New types of equipment included (1) a powered ramp plate for cleaning up coal spillage in front of a longwall face conveyor; (2) a motorized portable service machine to supply oil, grease, etc., to face equipment; (3) shortwall shearers for entry driving in conjunction with longwall mining; and (4) a battery-powered hour meter to tell how long a motor or a machine has been in operation. Schedule testing of longwall mining equipment was continued at increased levels, and a few approvals of 1,000-volt (nominal) face equipment have been issued or are in progress. Seven flame safety lamps from mines in which gas ignitions had occurred were investigated. All were found to be in safe condition. Research was continued to obtain basic information to facilitate analysis, testing, and design of intrinsically safe apparatus and instruments.

Eight approvals were issued under Schedule 25B to cover the integral roof drilling and dust-collecting systems of permissible machines.

Sixteen respiratory protective devices were approved under various Bureau schedules. Schedule 21B for filter-type dust, fume, and mist respirators was amended effective June 19, 1969.

SECTION 4. - EXPLOSIONS AND EXPLOSIVES

Methane and Dust Research

Dust Cloud Formation and Ignition

In studies to assess the effects of weak mine-gas explosions, aerodynamic characteristics of airflows in a small wind tunnel were measured with a hot-wire anemometer. Air velocities and turbulence intensities computed from boundary layer theory were in agreement with measured values. High-speed movies were taken of dust as it was being dispersed, and minimum air velocities for dispersion of variously shaped piles were measured. Dust beds were prepared from Pittsburgh seam bituminous coal, rock dust, and mixtures of the two; beds were also prepared from rock dust containing anticaking agents and from glass beads. A shear force apparatus was used to obtain a second measure of the force required to disrupt a dust pile. Empirical fits to wind tunnel and shear force data indicated that minimum air velocities were functions of the bulk density, shear strength, and length of the pile in the direction of the airflow. A fast-response weight cell was used to measure rates of dispersion of various dust piles.

A study was initiated to bridge the gap between small wind tunnel and large-scale experiments in the Experimental Coal mine. A 2-foot-diameter, 163-foot-long explosion tunnel was equipped for measuring significant characteristics of natural gas-air flames propagating through the tunnel, as well as the turbulence induced ahead of these flames.

High-pulse lasers were again used to electrostatically irradiate suspended particles. Experiments with individual particles of anthracite and bituminous coal showed a distinct difference in the ignition and combustion mechanisms of these two materials. A 60-micron bituminous coal particle irradiated with a beam intensity of 50 joules per square centimeter exploded and burned completely in less than 1 millisecond, whereas the same size anthracite particle broke into a few large fragments that did not appear to burn appreciably.

Experiments with irradiated individual magnesium particles (25 to 120 microns) in a stoichiometric methane-air mixture showed that the minimum radiant energy required to ignite the gas increased as the particle size decreased; this is opposite to the observed energy requirements for ignition of similar particles in air. The minimum laser radiation intensity necessary to ignite a 25-micron magnesium particle in air is much less than that required for such a particle to ignite a stoichiometric methane-air mixture. Ignition of the methane-air mixtures appears to occur in three stages: First, there is an almost explosive growth of the metal vapor cloud surrounding the particle. Next, a spherical metal vapor envelope surrounding the particle expands more slowly (at about 400 centimeters per second) to a diameter of about 0.4 centimeter. Finally, the radial velocity drops to a constant velocity of about 200 centimeters per second, corresponding to the combustion of the methane-air environment.

Minimum spark energies for ignition of coal dust dispersions in air were determined. The energy required to ignite a dust cloud was found to depend on the electric circuit characteristics to a much greater extent than is the case of gas systems. Under optimum conditions, the minimum stored energy required to ignite dispersions of minus 200-mesh Pittsburgh coal was about 15 millijoules, which when corrected for electrical efficiency corresponds to an ignition energy of about 10 millijoules.

A program was initiated to study the molecular aspects of the various chemical and physical processes of thermal ignition of combustible gases. Use is being made of recently developed light-scattering techniques based on the Brillouin and Rayleigh scattering processes. A bow shock developed in a supersonic nozzle is used to heat and compress the desired reacted mixture to a temperature near its thermal ignition value. Events behind the shock are monitored continuously by a special interferometric technique.

Quenching Ignitions and Explosions

Research was continued on developing a device to sense and quench a methane ignition at the face before appreciable flame or pressure develops. It consists essentially of an ultraviolet sensor and a pipe containing the quenching agent and a rupturing explosive charge. The disperser was shown to operate effectively when filled with Halon 1301. With this extinguishing agent, smaller explosive charges are required to rupture the tube, thus minimizing the accompanying noise. The modified quenching system was also used successfully to arrest a propagating coal-dust explosion at 75 feet from the face, compared with 400 feet without the quenching system (fig. 22).

Procedures were developed for sampling and analyzing toxic combustion gases (halogens and halogen acids) formed when Halon 1301 is used as a fire extinguishant. Threshold limit toxicity values of these gases range from 0.1 to 3 parts per million.

Explosion tests in the Experimental Coal mine showed that water inerting of mine roadways reduces the coal-dust-explosion hazard when coal dust deposits on the rib-roof surfaces are neutralized with dry rock dust. The floor should contain 26 percent or more water, and all coal particles should be wetted (fig. 23).

In other experiments at the Experimental Coal mine, to determine the explosibility of float coal dust deposited over rock dust, weak to strong explosions were readily obtained, depending on the loading density of the dust and the length of the explosion zone. In an 830-foot-long zone, a superficial deposit of 475 pounds of fine coal dust over 1,210 pounds of

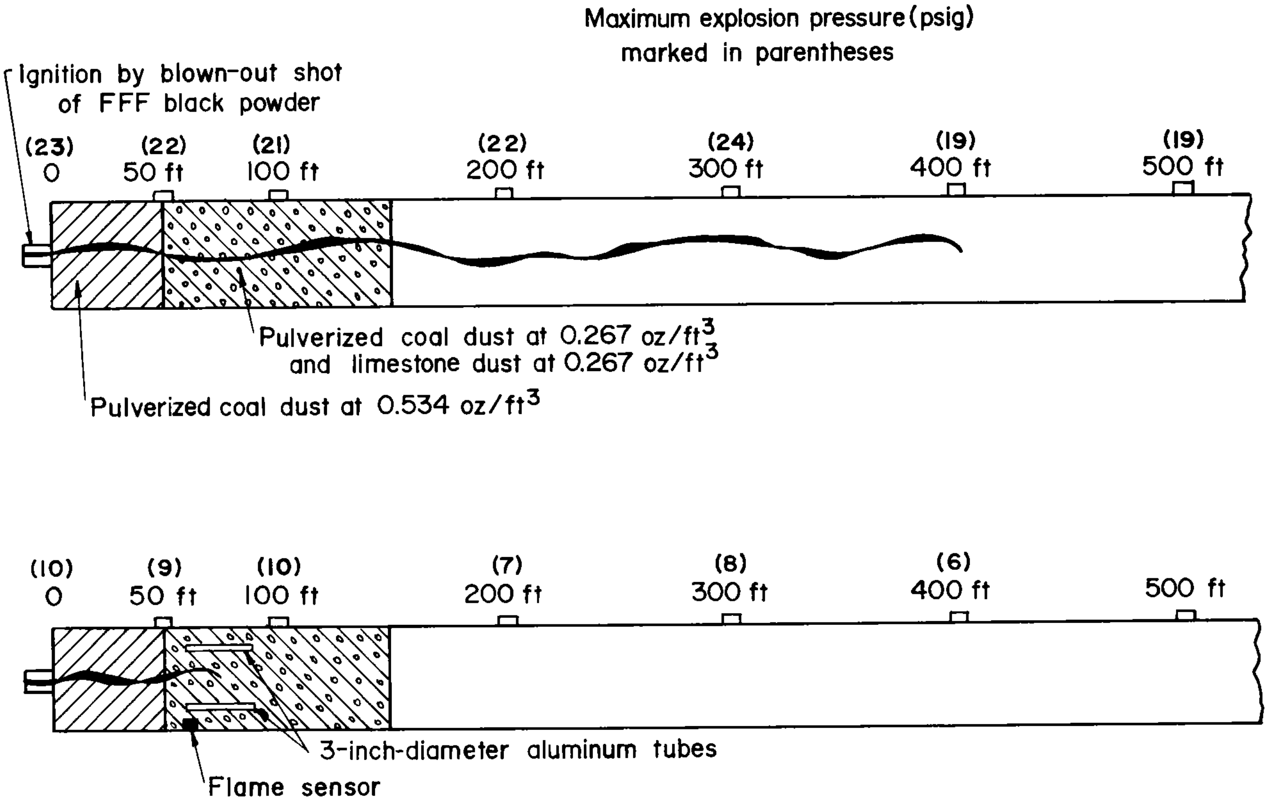

FIGURE 22. - Coal Dust Explosions Without (Top) and With (Bottom) Quenching Device.

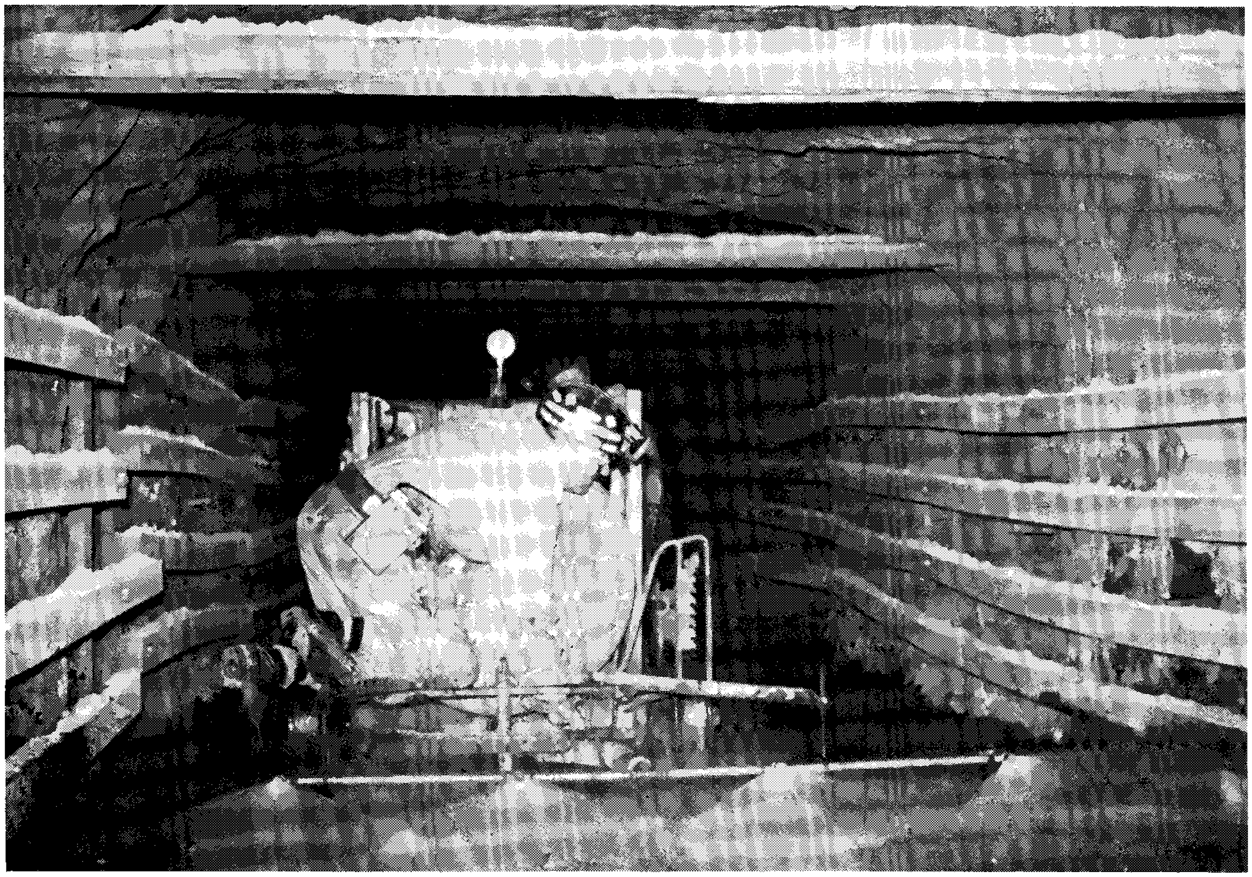

FIGURE 23. - Spraying Water on Coal Rubble on Mine Floor.

machine-distributed rock dust gave a strong dust explosion with supersonic flame velocity. The ignition source was a spark-initiated 1,300-cubic-foot mixture of 6 percent methane in air.

Measurements were made to determine quantitatively the rock dust/coal dust ratio and the rock dust/coal dust + methane ratio required to inhibit flame propagation in dust clouds of known concentrations. To eliminate quenching by the walls of the 15-centimeter (ID) by 3.6-meter-long duct, the walls were heated electrically to at least 100° C (212° F). The experimental conditions were chosen to give a well-defined coal dust concentration which remained uniform throughout the dust cloud as the flame traversed it. In previous experiments on a larger scale, there had been some uncertainty on these points. With the duct walls heated to 100° to 200° C (212°-392° F), rich coal dust-air mixtures required about 44 percent rock dust to inhibit flame propagation; with the walls at 250° to 300° C (482°-572° F), about 53 percent rock dust was required. With the stoichiometric coal dust-air mixture, about 40 percent rock dust was required to inhibit the flame at about 200° C wall temperature. The addition of methane to the coal dust cloud reduced the ability of rock dust to inhibit flame. At about 200° C, 1 percent methane raised the rock dust requirement for the rich coal-dust mixtures to about 59 percent;

2 percent methane raised the rock dust requirement to about 67 percent. Results of experiments now in progress indicate that a number of commercial chemical extinguishants inhibit flame more effectively than does rock dust.

Mine Explosives

Permissible Explosives

The program for including a minimum of 10 percent sodium chloride in all American permissible formulations was successfully completed when a company (the last) made the necessary addition to all six of its name-brand permissible explosives.

Incendivity of Permissible Explosives

Twenty-three granular and four gelatinous permissible explosives were evaluated for incendivity in a mixture of 4-percent natural gas and 0.2 ounce per cubic foot of coal dust predispersed in air. The gelatinous explosives were found to present a considerably greater ignition hazard, with charge weights for 50-percent ignition probability ranging from 86 to 660 grams, as compared with 257 to 945 grams for the granular formulations.

Evaluation of Blasting Caps

Evaluation of a variety of electric blasting caps showed that the igniting action of these devices toward an 8-percent natural-gas-in-air atmosphere was much higher than that of electric blasting caps investigated in a much earlier study. This is attributed to changes in composition of the base charge over the years.

SECTION 5. - COAL STORAGE, PREPARATION, AND TRANSPORTATION

Consumption of lignite and subbituminous coals in the United States is increasing, and most of the present production is used to generate electric power. Both of these fuels are utilized essentially as-mined, without beneficiation; preparation ordinarily consists principally of crushing and screening. Several mines blend coal from different locations within the mine to control the composition of the ash. Such practices and possible future processing involving both washing and drying to alter ash composition and to upgrade low-rank fuels are described in a paper presented in 1969.[25]

Coal Drying

Lignite Dryer Development

Future processing of lignite to increase its fuel value or to improve its burning characteristics may require a drying step; therefore, efforts are

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[25] Ellman, Robert C., and John W. Belter. Preparation Practices in the Lignite and Subbituminous Coal Industry. Preprint Paper No. 69-F-362, Fall Meeting SME, Salt Lake City, Utah, Sept. 17-19, 1969, 15 pp.

underway to develop a cheap and dependable system. A pulse-jet combustion system has received considerable study because it offers lower capital costs (simple construction), heat for drying, and power to circulate the gas. Under a cooperative contract, a semicommercial size pulse-jet engine with a heat release capability of 25 million Btu per hour was installed at the coal-drying plant of a North Dakota company and was operated on partly dried lignite dust from the mine-operated dryer. Although the fuel was relatively coarse, operations were maintained by adding small quantities of an oil or propane to promote ignition. After about 20 hours of operation, slag buildup in the combustion chamber forced a shutdown. The refractory lining of the chamber did not erode and mine personnel did not regard the engine noise as unacceptable, although no effort was made to muffle or suppress the sound. A paper was presented describing experience in lignite-fueled, pulse-jet engines.[26]

A dual pulse-jet unit is being tested as a potential means of conducting shock tube chemical reactions. When operated tailpipe-to-tailpipe, each pulse jet fires alternately with mutual reinforcing of the pressure waves. Lignite activation and gasification have been achieved and other applications are being considered.

Ultrasonic Drying

Ultrasound as an aid in the evaporative drying of fine coal was investigated with the apparatus shown schematically in figure 24. Pittsburgh seam bituminous coal containing about 10 percent moisture and ground to 70 percent through 200 mesh was fluidized with air heated to 300° to 400° F. Sound at frequencies of 17, 20.2, 35.5, 65, and 93 kiloHertz and amplitude of about 135 decibels, when applied above the bed, increased the drying rate by almost 47 percent over that achieved without application of sound, and by 31 percent over "no-sound" drying when applied below the bed. The increase in drying rate occurred during the normally constant rate period; the higher increases in drying rate were effected by the lower frequencies.

Coal Cleaning

Washability Characteristics Survey

Appalachian Coals

Another report was issued on the washability characteristics of the principal coals of the Appalachian region.[27] To date, reports are available covering 63 Appalachian region counties (fig. 25). Analyses were completed of 21 coal samples from Ohio, and an additional 20 samples were collected from the Ohio coalfield. Counties sampled are also shown in figure 25.

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[26] Belter, J. W., L. Dockter, and R. C. Ellman. Operating Experience With Lignite-Fueled Pulse-Jet Engines. Preprint Paper No. 69-WA/Fu-4, Winter Ann. Meeting of ASME, Los Angeles, Calif., Nov. 16-20, 1969, 5 pp.

[27] Zeilinger, J. E. Preparation Characteristics of Coal From Beaver and Lawrence Counties, Pa. BuMines Rept. of Inv. 7324, 1969, 23 pp.

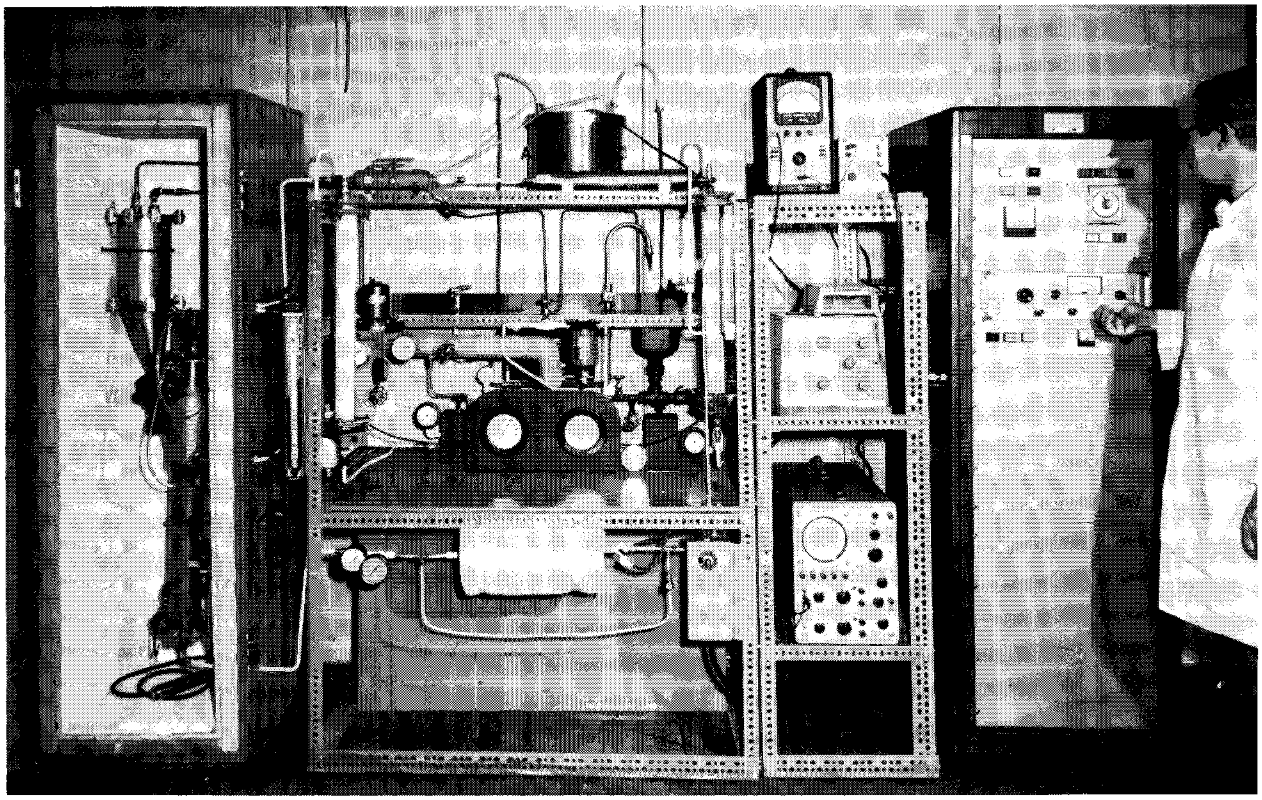

FIGURE 24. - Studying Ultrasonic Energy for Drying Finely Divided Coal.

Pennsylvania Anthracite

No additional anthracite samples were processed because the study was discontinued. A report is being prepared covering the washability of the Kidney Seam, Northern Anthracite Field.

Coal Cleaning Methods

Froth Flotation

Current froth-flotation techniques are not selective enough to substantially reduce the sulfur content of finely sized coals. Tests are in progress with a laboratory-scale, froth-flotation cell to determine the effectiveness of selected reagents to depress pyrite, and thus separate it from the coal. These tests have demonstrated that the presence of complex metal ions formed by the dissolution of multivalent metal salts in water are potential pyrite depressants. Because various metal salts are subjected to different complexing in aqueous solutions, their ability to depress pyrite during froth flotation was found to be a function of salt concentration and solution pH. Work was continued.

Statistical analyses of froth-flotation operating variables was continued with the primary purpose of assessing, by a statistically designed program,

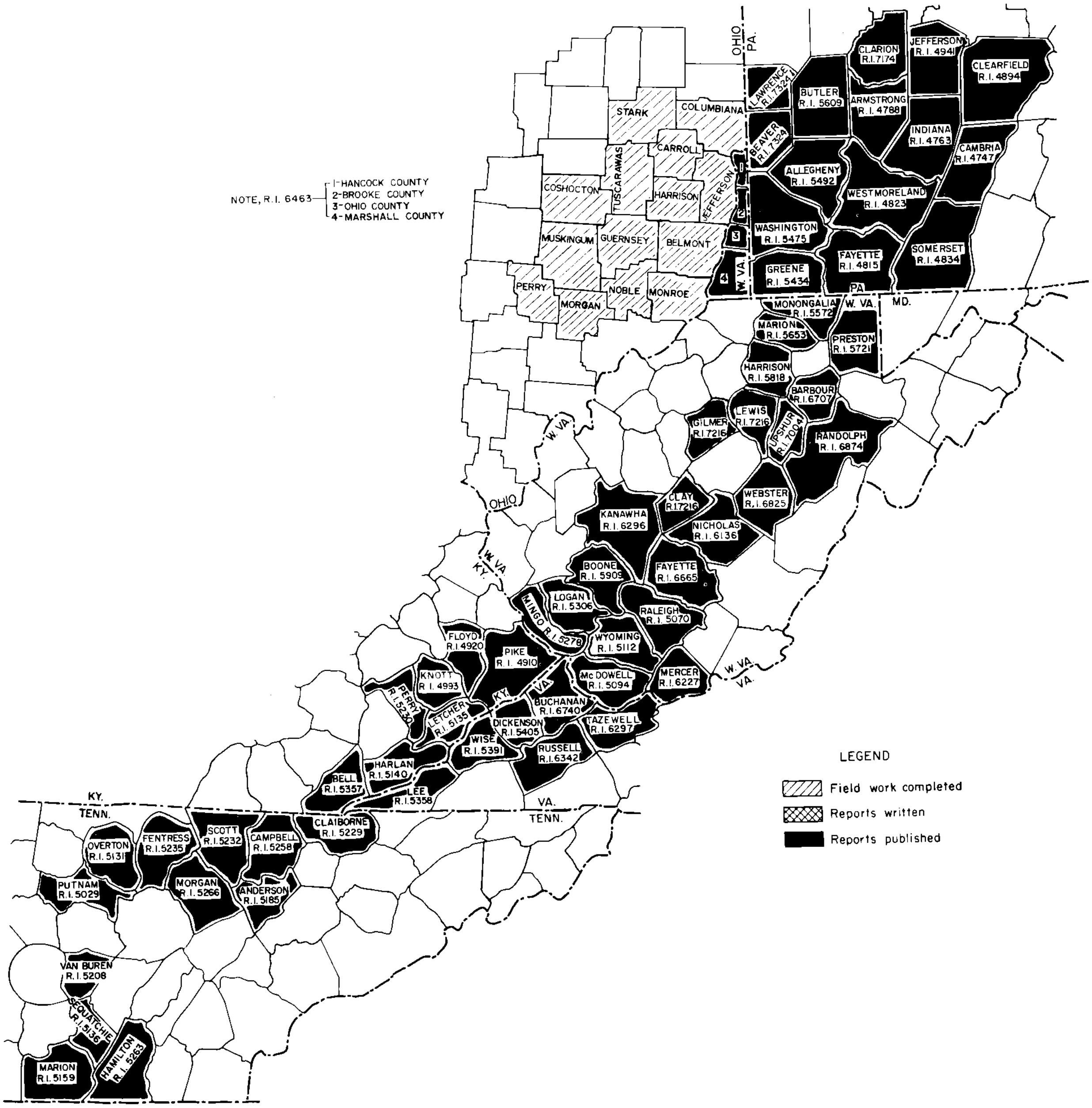

FIGURE 25. - Washability Reports, Appalachian Coals Through 1969.

the effects of the principal operating variables that affect the coal flotation process. Objectives of the study are to help reduce stream pollution and to increase the quantity and quality of the coal product. Computer analyses and statistical evaluation of pilot-plant test data were completed from which prediction equations were derived for six coals based on the dependent variables of clean-coal yield, ash content, and power input. These equations make possible an evaluation of the main and second order interaction effects of the independent variables, and also those effects due to differences in the independent variables.

Dense-Medium Washers

Performance data for the major types of equipment in American coal preparation plants can assist in the design and operation of coal-cleaning equipment. Surveys of concentrating tables, sand cones, and dense-medium coarse-coal vessels were completed before 1969. A fourth phase of the survey--a study of commercial fine-coal dense-medium cyclone washers--was completed in 1969. Dense-medium cyclones, in general, effected excellent separations of coal from impurities.

Investigation was also concluded of the effect of magnetite size on sharpness of separation in a dense-medium cyclone coal washer. In this study, all of the operating variables were held constant while the size consist of magnetite in the media was varied from 76 percent minus 325 mesh to 98 percent minus 325 mesh. Error curves used to evaluate the data demonstrated, in most instances, that media composed of the finer magnetite produced the sharpest separations at constant separating densities. A predictable relationship was established between the separating gravity and the medium gravity.

Sodium Removal by Ion Exchange

The feasibility of removing sodium from lignite by ion exchange methods is being investigated. Ion exchange methods have been found to remove virtually all sodium from the lignite, but little is known about the kinetics of this process. Laboratory studies include analyses of the effect of ion concentration, time, and particle size in a countercurrent process. Dilute solutions in an ion exchanger were found to be surprisingly effective in sodium removal; the exchange rate was practically instantaneous for pulverized sizes, but is a function of size. A practical process will depend on the relationship between exchange rate, size, and exchange-media cost.

Quality Control

Work was continued on continuous methods of determining the moisture and sulfur content of coal to facilitate production of fuels meeting industrial requirements and air pollution regulations (fig. 26). In 1969, a prototype moisture meter utilizing radioisotopes was installed and operated at the U.S. Steel Corporation's coal preparation plant at Robena, Pa. (fig. 27). The device continuously recorded the moisture content of 5- to 10-ton-per-hour streams of coal within 0.2 percent of values determined by conventional analysis, and operated for 11 months without electronic failures. Several instrument companies have expressed interest in manufacturing a commercial meter.

Also developed and tested at Robena was a device that utilizes gamma rays from radioactive sources to determine the bulk density of coal in pounds per cubic foot. Control of bulk density in coke plants is essential to production of high yields of metallurgical coke. The gamma ray meter has given bulk density readings within 0.2 pound per cubic foot of actual values.

Work was continued on a sulfur meter (for coal) that utilizes neutron-gamma reactions. Californium-252 has been obtained from the U.S. Atomic

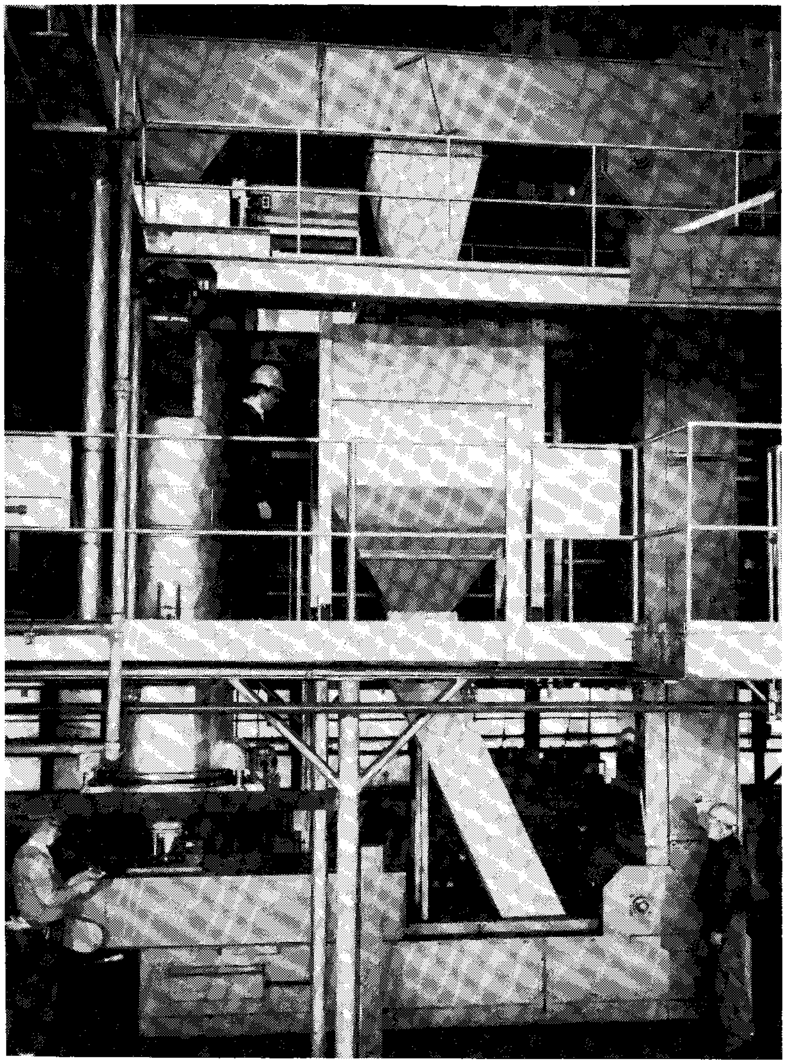

FIGURE 26. - Coal Conveying System Used To Develop Nuclear Methods of Coal Analysis.

Energy Commission for use in developing this meter. Californium-252 produces a large number of relatively low-energy neutrons with less interference radiation than is accompanied by other neutron sources. The material shows promise of wide application in process control.

Coal Pipelining and Handling

Hydraulic Transportation

Hydraulic transportation of mined materials for long distances continues to grow. Notable new entries in the field are the 53-mile-long, 9-inch pipeline carrying iron ore slurry over the rugged terrain of Tasmania, Australia, and the 273-mile, 18-inch coal pipeline from northern Arizona to southern Nevada. In both instances, feasibility studies revealed that pipelining was cheaper than alternate transportation methods. Despite these recent successes, a continuing inability to predict accurately the head loss under given conditions is a factor impeding the development of this type of system. Head loss determines power requirements, and the latter is the main factor in establishing pipeline operating costs. Investigations were continued, therefore, in an effort to reduce and predict head loss in slurry pipelining.

Results were published of tests with a 1-mile-long, 6-inch-diameter pipeline.[28] Experiments were also conducted with an 80-foot-long, 1-inch-diameter laboratory pipeline to determine the effect of geometric shape on head loss and delivered-solids concentration. Head loss curves for pipes of various cross-sectional shapes are presented in figure 28. A rectangular pipe with a

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[28] Fowkes, R. S., and G. A. Wancheck. Materials Handling Research: Hydraulic Transportation of Coarse Solids. BuMines Rept. of Inv. 7283, 1969, 36 pp.

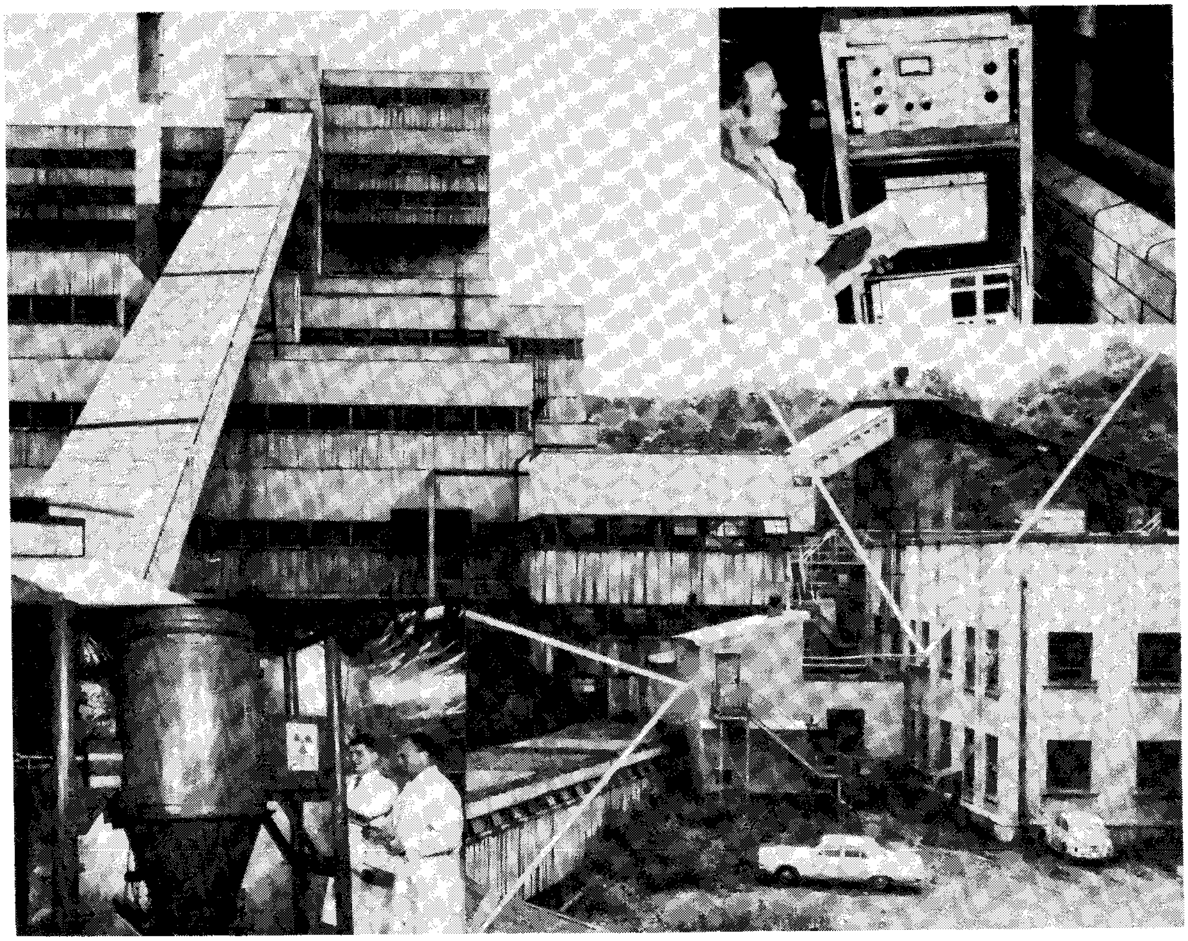

FIGURE 27. - Coal Preparation Plant, Robena, Pa.

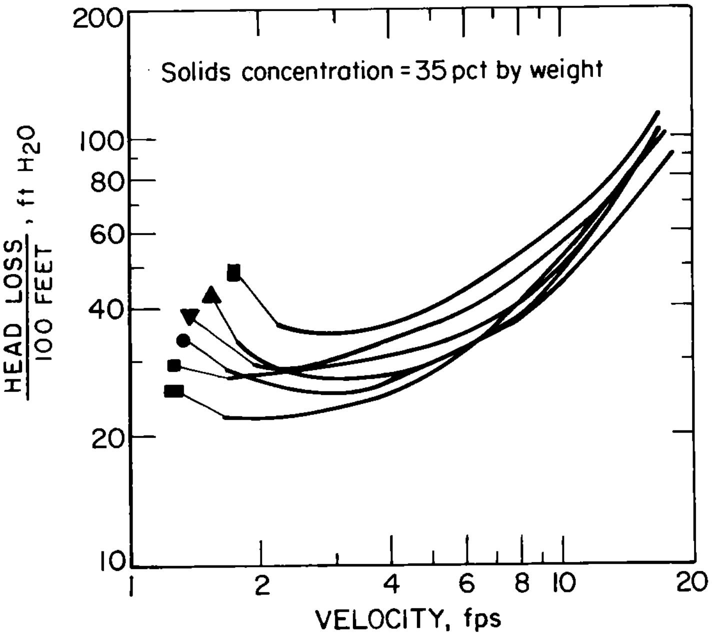

FIGURE 28. - Head Loss Curves of Pipes of Different Cross-Sectional Shape (Out Solid Concentration of 35 Percent by Weight).

2 to 1 width-height ratio reduced the head loss approximately 20 percent, enough to suggest that pipe shape is a factor to consider in design.

Coal Handling

Solids Conveyor

Mining companies critically need continuous materials-handling systems that are modular so that they can be lengthened quickly, be powered at intervals along their length, can traverse corners, and will not malfunction from misalinement. To meet these requirements, a novel conveyor is being investigated on which material is moved along a flexible surface that is undulated by means of sequential vertical-belt displacement. A test loop was completed for studying the performance characteristics of such a conveyor. Essential features of the conveyor are illustrated in figure 29.

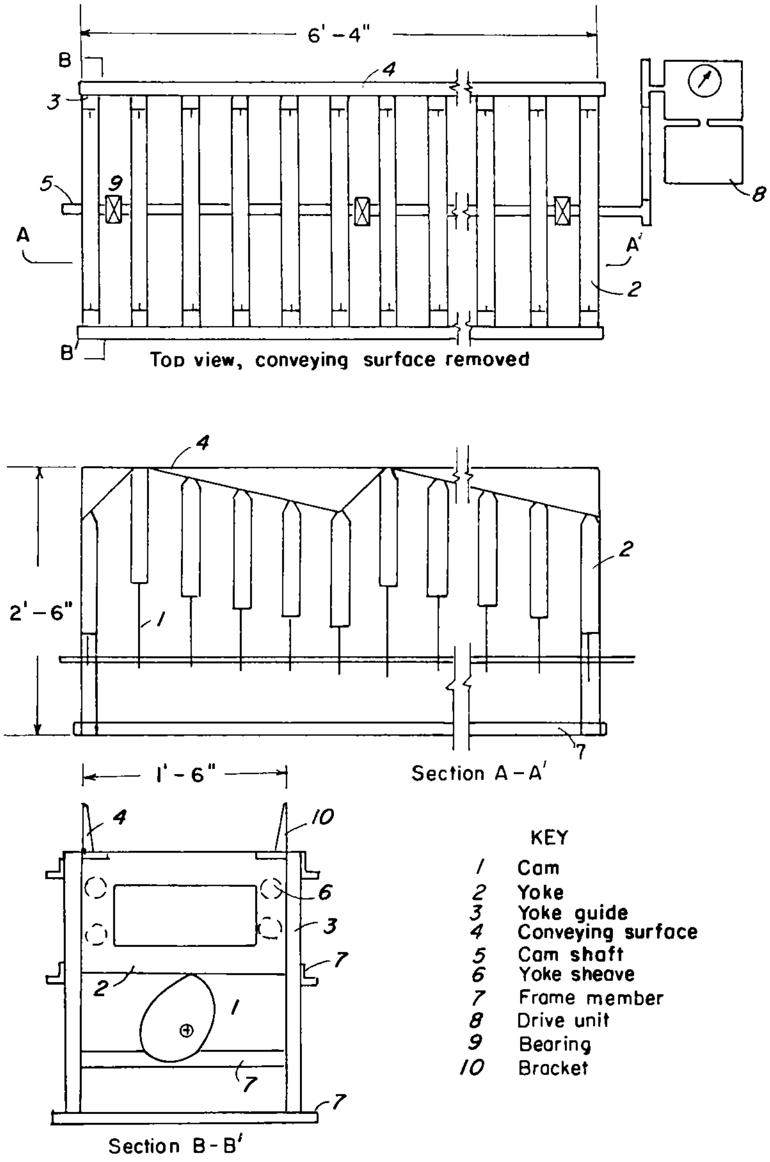

FIGURE 29. - Undulating Type Solids Conveyor.

Mechanism of Coal Packing

Packing can interfere with and even stop the flow of coal in coal-handling systems. Conditions that cause the coal to pack are being simulated in an attempt to obtain information on the mechanical properties of packed coal and the energy required to redisperse the coal. Of major interest is shear strength and the variables likely to effect it. These include particle size, moisture content, surface properties, compaction pressure, duration of pressure application, and vibration during compaction. A literature search was completed and a test system was designed for use in measuring the shear strength of coal packed under various conditions.

SECTION 6. - ELECTRIC POWER GENERATION FROM COAL

Coal-Burning Powerplants

Boiler Tube Slagging and Corrosion

Field Tests, Lignite-Burning Powerplants

Close contact was maintained with the larger lignite-fired powerplants to determine the extent and nature of problems relating to the combustion of lignite. Work was carried on with the Basin Electric Power Cooperative in a program to try to correlate ash analyses (chemical) and ash-fusion data with the extent of superheater and reheater fouling in their 200-megawatt pulverized fuel-fired boiler at Stanton, N. Dak. During plant operations, officials had noted generally good correlation between boiler fouling and the sodium content of the coal. In a test, however, the fuel quality remained consistent, yet boiler fouling appeared to vary significantly. The results demonstrate the problem of full-scale boiler performance evaluation and indicate that studies of boiler fouling must take into account such factors as boiler load schedule, soot blowing schedules, time since last cleaning, and on-line hand-lancing schedule.

Tube Fouling, Lignite, and Western Coals

Pilot-plant studies of problems relating to combustion of lignite and other Western coals in utility powerplants were continued. Problems receiving most attention are ash deposition on boiler heating surfaces and electrostatic precipitation of fly ash from boiler flue gases. More than 50 tests were made in 1969 with a 75-pound-per-hour pilot-scale furnace (fig. 30) to evaluate the fouling tendency as a function of coal ash analysis and other parameters.[29] Coals from nine States were evaluated, including 25 lignites, three subbituminous coals, and four bituminous coals. One of the principal conclusions was that by far the most important single factor in the relative fouling potential of these coals is the sodium content of the ash (fig. 31). The fouling rate increases rapidly with sodium content up to about 8 to 10 percent Na_2O in the ash, after which further increase in sodium (up to 25 percent) has little effect. A second important finding is that a North Dakota lignite having 40 percent CaO in the ash fouled significantly less than lignites containing one-half as much CaO in the ash (fig. 32). Next to sodium content, the most important factor in ash deposition rate is the flue gas temperature at the deposition area. Other factors such as excess air, fuel moisture content, and fineness of pulverization have little effect on ash-deposition rates.

In one series of tests, two different bituminous coals were burned in the test furnace for comparison with typical "high-fouling" and "low-fouling" lignites from North Dakota. One of the bituminous coals was from central

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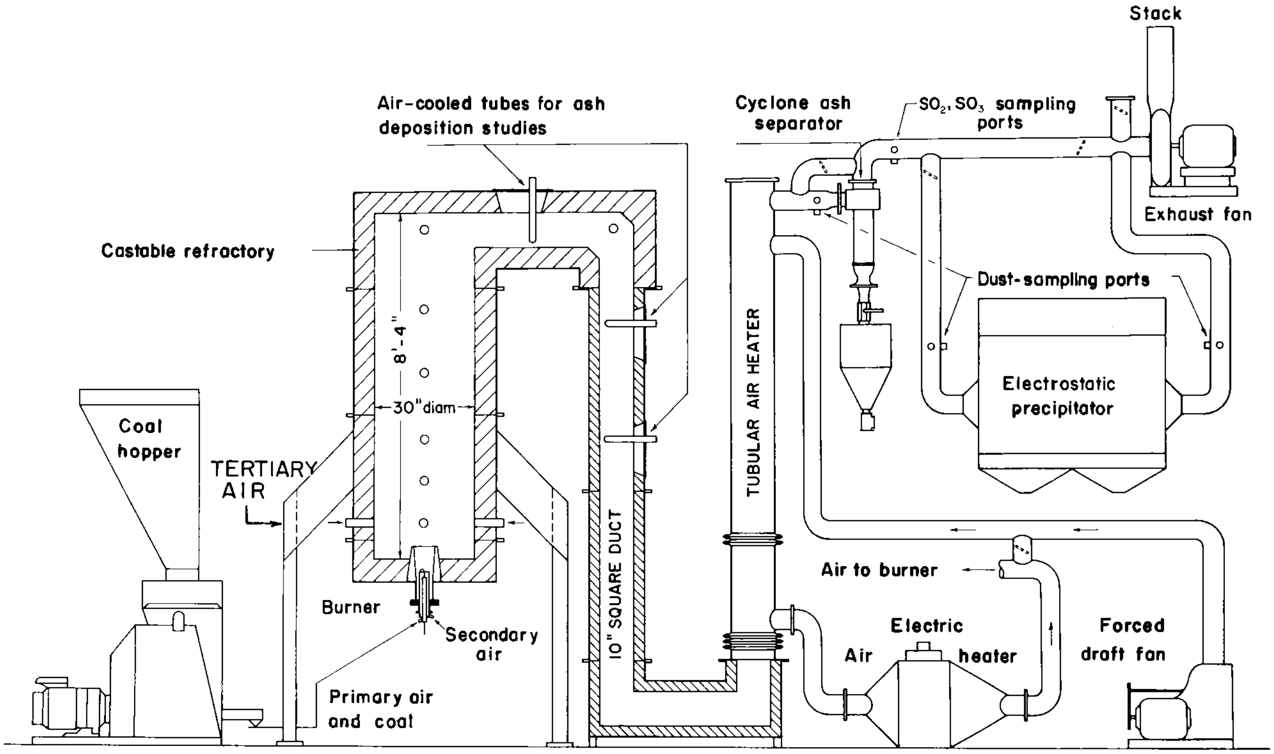
[29] Gronhovd, G. H., W. Beckering, and P. H. Tufte. Study of the Factors Affecting Ash Deposition From Lignite and Other Coals. Preprint Paper No. 69-WA/CD-1, Ann. Meeting of ASME, Los Angeles, Calif., Nov. 16-20, 1969, 9 pp.



75 LB/HR PULVERIZED COAL-FIRED FURNACE

FIGURE 30. - Flowsheet of Furnace for Ash Fouling and Air Pollution Studies.

Illinois and was considered to be high fouling; the other coal came from southern Illinois and was rated low fouling. Both of the Illinois coals had considerably higher ash contents and 200° to 300° F lower ash-fusion temperatures than the lignites. Also, the chemical compositions of the bituminous coal ashes were much different than for the lignites: higher in silica, iron, and alumina content and lower in calcium and sodium content. The rate of ash buildup on the tubes for the high-fouling bituminous coal was found to be less than half that of high-fouling lignite. These results indicate that the problems involved in burning high-fouling North Dakota lignites are different from those for high-fouling bituminous coals and suggest that equipment manufacturers may not have given enough attention to designing boilers to burn the former. Results with low-fouling Illinois coal were similar to those from low-fouling lignites, but when sodium bicarbonate was added to this Illinois coal to increase its sodium level to about 5 percent Na_2O in the ash (similar to a high-fouling lignite), ash-fouling became extreme.

The test furnace is also being used to study the mechanisms of ash deposition in hopes of finding ways to reduce fouling. One approach has been to operate the furnace on natural gas and inject into the combustion zone one or more chemical compounds containing the elements found in coal ash. It is hoped that by reducing the number of compounds present in the combustion chamber, the role of each element or compound in contributing to deposit formation may be more closely defined.

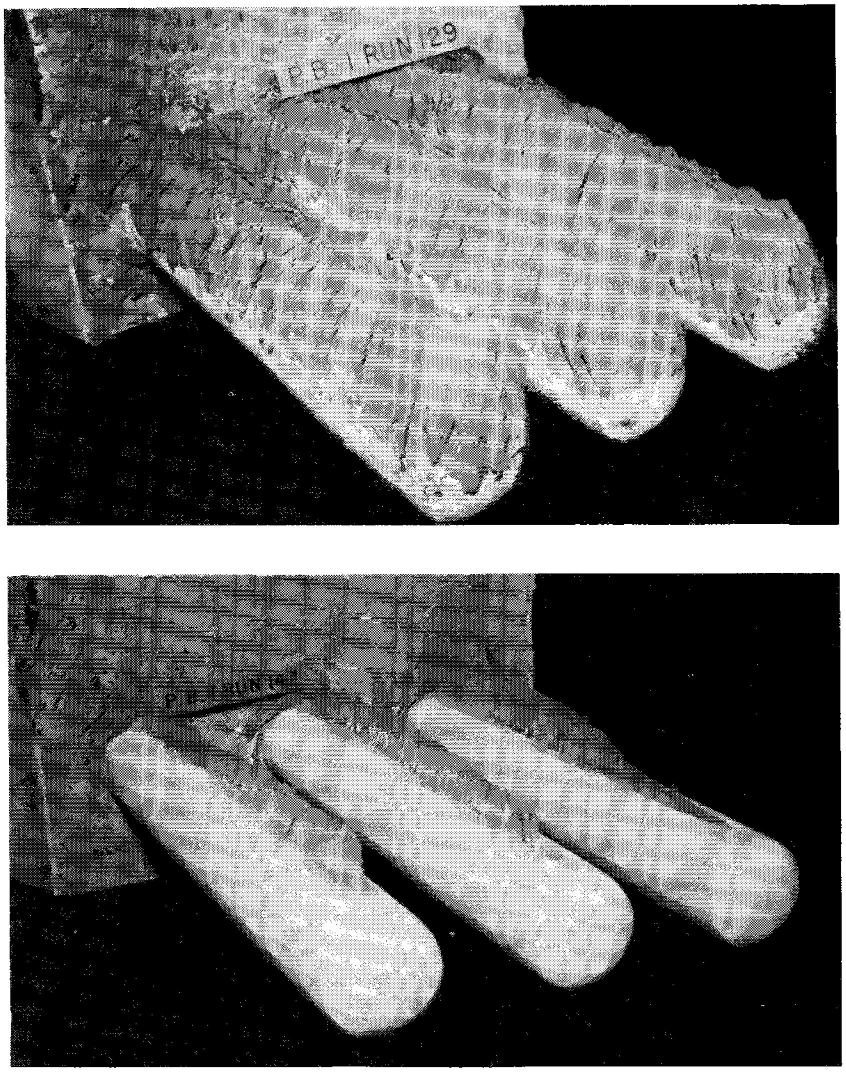


FIGURE 31. - Ash Deposits From High-Fouling (Top) and Low-Fouling Lignites (Bottom).

Study was begun of the factors affecting electrostatic precipitation of fly ash from lignite and other Western coals. Most of the present commercial electrostatic precipitators perform at below design efficiencies when cleaning combustion gases from low-sulfur Western fuels. Tests were conducted with a pilot-scale electrostatic precipitator on fly ash-laden gas from a Montana subbituminous coal to determine the effect on ash-removal efficiency of varying the temperature over the range 250° to 450° F. Preliminary results indicate little effect of gas temperature on precipitator efficiency, contrary to some test results obtained by a utility company on a full-scale precipitator.

Ash Composition of Western Coals

Western coals and lignite continued to be surveyed for composition, particularly ash, for use by producers and consumers. Data obtained on survey samples and on modified ashes prepared in the laboratory were used to derive a series of equations with which to predict ash-softening temperature. The results are being prepared for publication.

Basic Research on Coal Ash

Microprobe examination by very slow scan was conducted on a sample of high-sodium lignite (embedded in epoxy resin) for silicon, aluminum, iron, calcium, magnesium, sodium, and sulfur. The results show a very uniform distribution of all these elements except silicon. This indicates that most of the inorganic material in lignite is bound to the organic portion of the coal, and supports findings from previous float-sink tests. Silicon occurred as

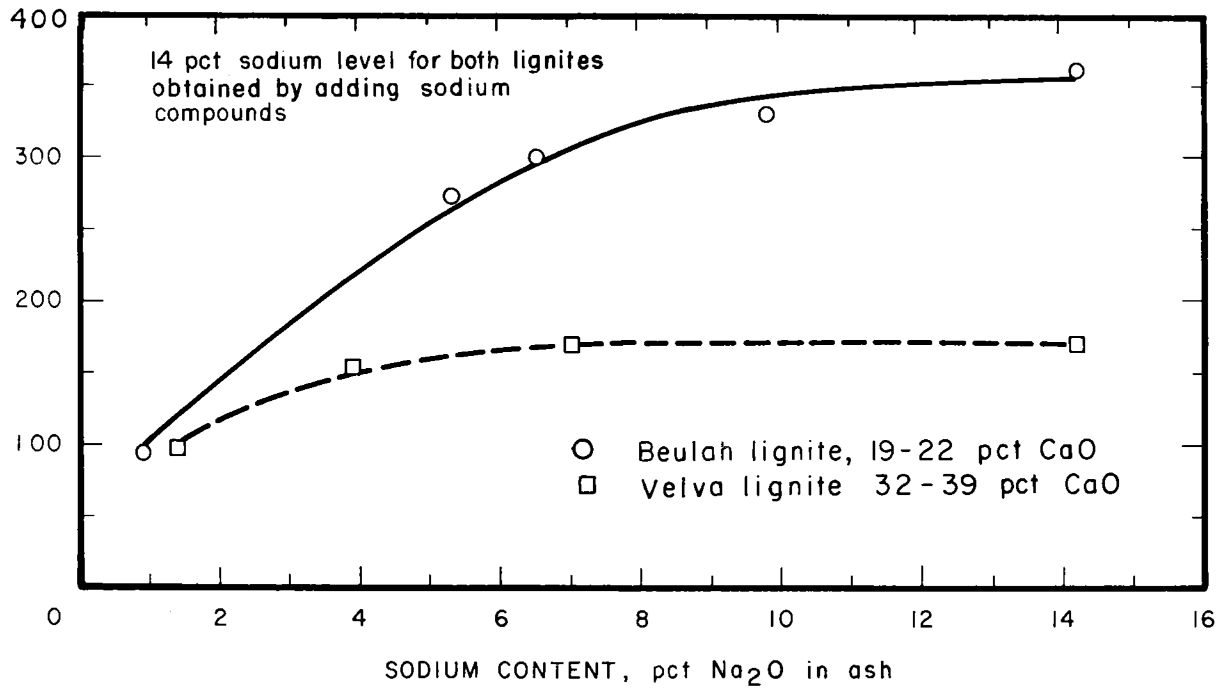


FIGURE 32. - Ash-Fouling Rates of High- and Normal-Calcium Lignites as a Function of Sodium Content.

small, discrete particles, usually quartz but in some cases complex silicates. Examinations of other coals from different localities have shown occasional inclusions of pyrites and calcium sulfate (gypsum), but no concentration of minerals in which either sodium or potassium is a major constituent. Infrared photographs of lignite or leonardite seams, such as figure 33, show mineral inclusions as light areas or bands.

A paper was published on sample preparation and examination of lignite ash by electron microprobe.[30]

A computer program was prepared to improve X-ray fluorescence analysis for the ten major elements generally reported in ash and deposit analysis. This has greatly increased the reliability of the procedure and reduced the minimum sample requirement to about 0.1 gram for a satisfactory analysis. A paper on X-ray examination of coal and coal ash was presented at the Bureau of Mines-University of North Dakota Symposium, May 1969.

In the study of low-temperature oxidation by microwave-activated plasma, samples of high-sodium lignite were treated at two Bureau laboratories and Tracerlab, Inc. Diffraction patterns of the residues indicated small amounts of sodium nitrate in the sample analyzed by a Bureau laboratory on a unit that operates at 150° C (302° F); apparatus at the other two laboratories operate

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[30] Fowkes, Walter W., Thomas D. Wilhelm, and Wayne R. Kube. Sample Preparation and Electron Microprobe Examination of Lignite Ash Deposits. Metallography, v. 2, No. 2-3, 1969, pp. 209-225.

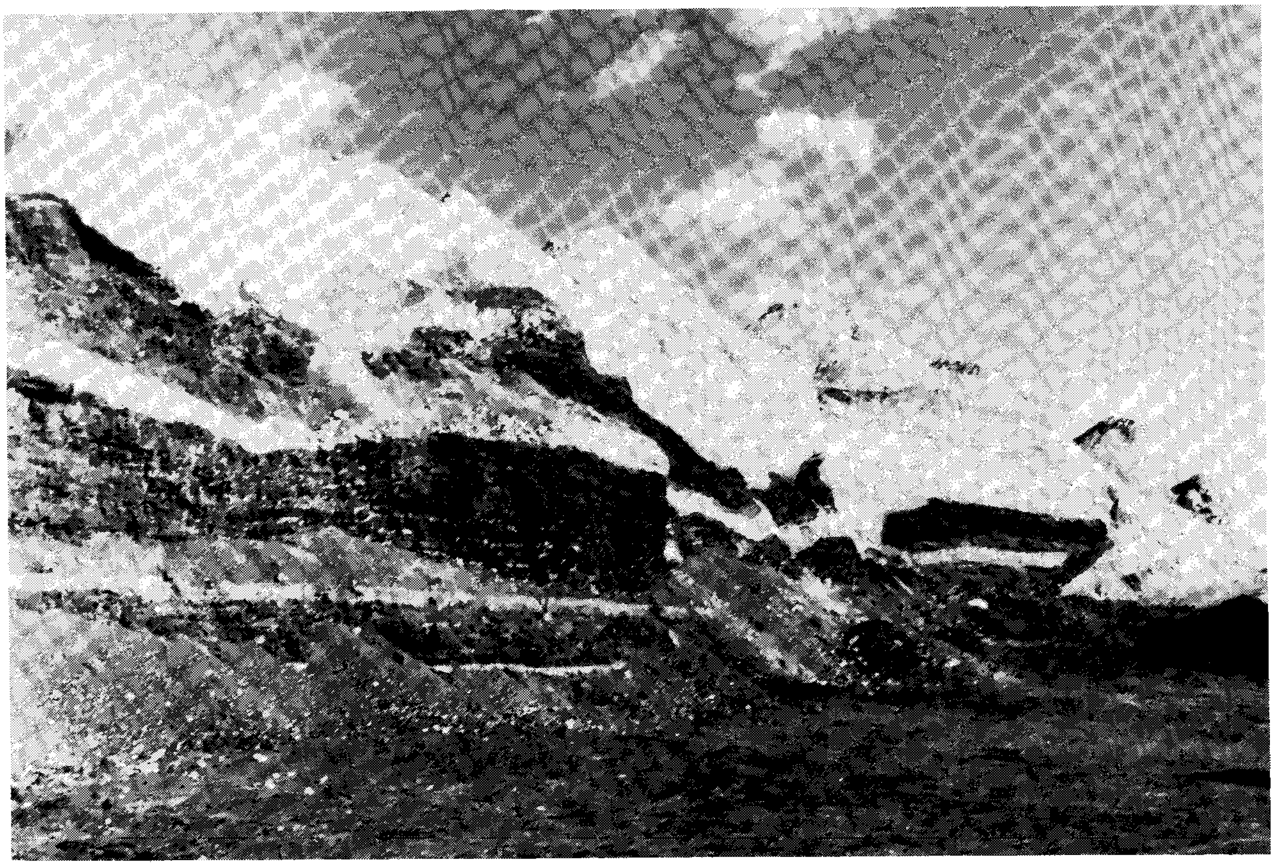

FIGURE 33. - Shale Partings and Mineral Inclusions (Bands of Light Material) in Lignite Seam, Southwestern North Dakota.

at near 400° C (752° F). The sodium salts of beta-naphthol and 1-naphthoic acid were oxidized under the same conditions used for lignite, and the residues found to be pure sodium nitrate.

A Bureau of Mines-University of North Dakota fellowship project[31] was completed on the sulfur dioxide absorption characteristics of lignite fly ash. Ash composition, exposure, time, and temperature all significantly affected the amount of sulfur dioxide absorbed. Also, fly ashes relatively high in calcium and sodium absorbed greater quantities of sulfur dioxide. Compared with alkalized alumina, lignite required higher temperatures to absorb comparable quantities of the air pollutant. The temperatures required to effect appreciable absorption of sulfur dioxide by lignite fly ash make commercial usage unattractive unless higher ash loads in the boiler can be tolerated.

Research was started on the characteristics of coal ash that contribute to deposition and corrosion inside pulverized-coal-fired boilers. Studies are

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[31] Lukes, Ellen Bernard. Removal of Sulfur Oxides From Synthetic Flue Gas by Lignite Fly Ash. M.S. Thesis, University of North Dakota, Grand Forks, N. Dak., 1969, 47 pp. (Available from University of North Dakota library.)

being conducted with a laboratory-scale furnace in which ash constituents are injected through a flame onto tubes in a synthetic flue gas atmosphere. Use of radioactive sulfur-35 is planned to study the chemical reactions that occur as the ash constituents move through the flame to the tube, and to thereby establish which constituents in the ash are chiefly responsible for deposition. Aerodynamic studies of the tube are also underway to obtain information about ash impingement (fig. 34). Ultimately sought is a relation between ash composition and fouling tendency, for a large variety of coals, that can serve as a guide in coal choice and boiler operation.

Combustion Research

Char Combustion

Char combustion processes are not well developed, primarily because char is not produced commercially. Char availability may increase significantly during the coming decade, however, if new processes for coal carbonization, gasification, and liquefaction move successfully into commercial practice. Research is needed on char handling, shipment, and storage as well as char combustion.

Operations were begun in 1969 with a 500-pound-per-hour pulverized-coal-fired furnace that simulates the performance of industrial steam-generating furnaces. The combustion characteristics of chars is receiving initial study. Chars to be studied include high-volatile B bituminous made in the Bureau's entrainment carbonizer. Chars for the combustion tests will have selected properties, and other high- and low-rank coals may also be processed.

Fluid-Bed Combustion

Fluid-bed combustion is being investigated as part of a program sponsored by the National Air Pollution Control Association. Reduction of fireside corrosion and deposition, suitability for high-ash, high-moisture coals without

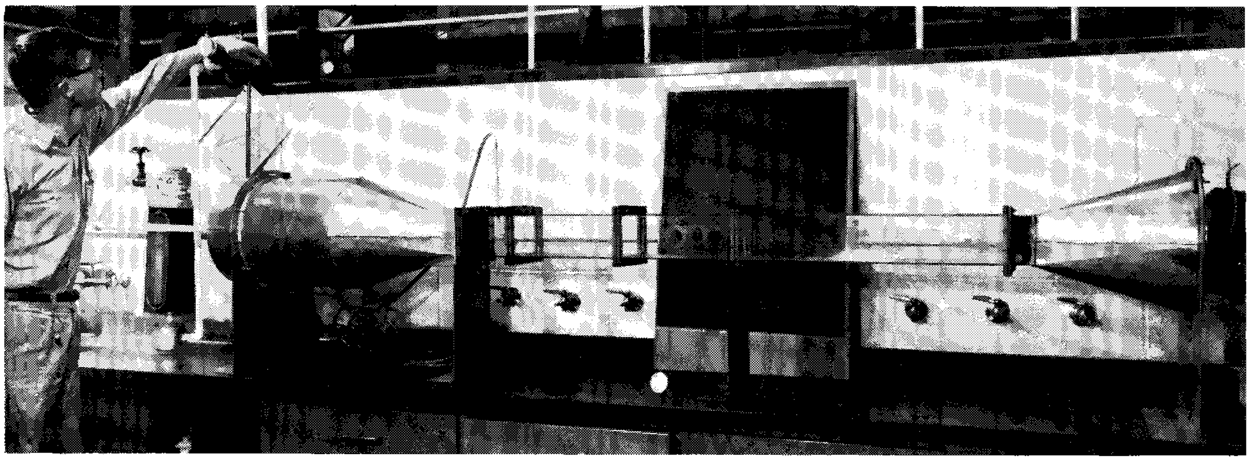

FIGURE 34. - Studying Coal Ash Characteristics That Induce Boiler Tube Deposition and Corrosion.

special preparation, and adaptiveness to the use of additives for sulfur dioxide removal are among the possible advantages of the method. The combustibility of various U.S. coals by the fluid-bed method is being determined, and tests are being conducted with each coal to establish the highest practicable temperature at which the fluid bed remains free from clinker formation. Also to be investigated is boiler-tube performance and heat transfer from the fluid bed to steam flowing inside the tubes.

Fuel Engineering Service

Onsite field tests of boilers and auxiliary equipment were conducted upon request by other Federal agencies. In addition, Federal agencies continued to avail themselves of the Bureau's consulting services related to fuels combustion.

A series of efficiency and capability tests were conducted on an oil-fired boiler of new design and its auxiliary dust collector at the National Institutes of Health, Bethesda, Md. Adjustments were made that markedly improved the overall operation of the boiler and auxiliaries.

Consulting services were provided for the Department of the Air Force in the modification of combustion equipment at Andrews Air Force Base, Camp Hill, Md. In addition, a member of the Bureau's fuel engineering service staff was requested by the Department of the Air Force to serve on a panel that makes final selection of Air Force heating and power generating plants in the competition for the Air Force's coveted annual William H. Bordner Awards.

Boiler Water Service and Research

Thirty-four Federal installations were added in 1969 to the thousands that continued to use the Bureau's industrial water services. The boiler water service is designed to cut maintenance costs and improve fuel (coal) economy in generating steam for heat and power. The cooling water service helps to prevent corrosion, incrustation, and fouling in large air-conditioning systems. Both services are based largely on in-plant testing, with test kits and methods supplied by the Bureau on a reimbursable basis. Preventive-maintenance research is conducted in close coordination with the industrial water services, to guide the consultants to practical solutions of water-related troubles.

Nearly 108,000 test kits and kit components were distributed in 1969, a decrease of about 2 percent over the 1968 figure, and a Bureau laboratory that analyzes the waters submitted by plant operators to check in-plant testing processed 12,000 samples, 1,000 less than in 1968. These decreases reflect an ever-increasing operator competence, at least partly attributable to workshops conducted periodically since 1961 by the Bureau for utility plant supervisors. Six workshops were held in 1969, attended by 62 personnel from nine Federal departments and independent agencies.

The many miles of piping that return steam condensate to boiler plants of Federal heating systems are especially vulnerable to corrosion. Continued use was made of the Bureau's corrosivity testers in 1969, and 236 were returned for evaluation. Estimated conservatively, annual maintenance costs for repairing or replacing corroded return lines were at least $7 million less in 1969 than in the early 1950's.

Cooperation was continued with industry in an effort to improve technology and upgrade codes and standards. A Bureau representative served as a member of the Executive Committee of the Research Committee on Boiler Feedwater Studies and as Chairman of Performance Test Code Committee 19.11. Both of these are committees of the American Society of Mechanical Engineers. He also is Chairman of Subcommittee B-19.7 on Properties of Water and Water-Formed Deposits, American Society for Testing and Materials.

Coal Resources Surveys

Studies were made of factors pertaining to demand, supply, and costs of coal in relation to domestic and foreign energy demands. Strikes, imbalanced productive capacity, and other influences caused a production decline during the year, notwithstanding that demand continued upward, both at home and abroad, by a total of 20 million tons. Shifts in energy-consumption patterns and costs, including fuel imports, were analyzed in relation to their effects on coal production, distribution, marketing, and utilization. Attention was centered on steadily increasing steam coal requirements for power generation and on the potential effects of air pollution standards on coal demand. Survey data were reviewed on changes in mining methods and on productivity and capacity in the industry.

The survey of coal reserves economically amenable to surface mining was advanced towards its final stages. Information developed will identify the location and extent of strippable coal reserves, the areas in which future surface mining are most likely to occur, and, correspondingly, the areas most likely to pose significant surface-land reclamation problems. Also being determined are cost estimates for developing and operating representative strip mines.

A survey on underground mine haulage was begun to assist in developing haulage systems compatible with the high productive potentials of continuous mining machines and other highly efficient equipment for removing coal from the mining face.

A detailed investigation was virtually completed of coal resources in the Uinta region of Colorado and Utah, an area that includes 12 distinct coalfields. In addition to reserves, geologic characteristics, and minability, special consideration was given to water availabilities, a very important factor in western mining operations. Attention also was directed toward potential markets for coal from this region, and to alternate transportation methods from mines to consuming areas.

Increased power generation in the North-Central region of the United States has intensified interest in lignite combustion and the problems relating thereto. Certain lignite ashes have a greater tendency than others to deposit on furnace walls and fireside surfaces of boiler tubes, reducing coal-utilization efficiency and increasing maintenance costs. Ash deposition appears to be related to the sodium content of the fuel. To provide information for mine planning, specifically for scheduling production to deliver a uniform product, work was undertaken to develop a method of predicting the sodium concentration of lignite deposits in advance of mining. A method based on statistical techniques was developed using surface coordinates from which both the coalbed elevation and overburden are predicted and a pattern of sodium values computed over the area under investigation. From this pattern is constructed a contour map representative of sodium distribution in the deposit. An isogram map for sodium concentration distribution is shown in figure 35. These data suggest that the high-sodium areas may be localized; a more extensive study is needed to determine whether these high-sodium areas are randomly distributed in the deposit. Also, the properties of the overburden (such as permeability) and characteristics of the coalbed (such as thickness and relative position of partings) need to be observed and measured to establish the effect of variables vis-a-vis ash deposition and sodium distribution.

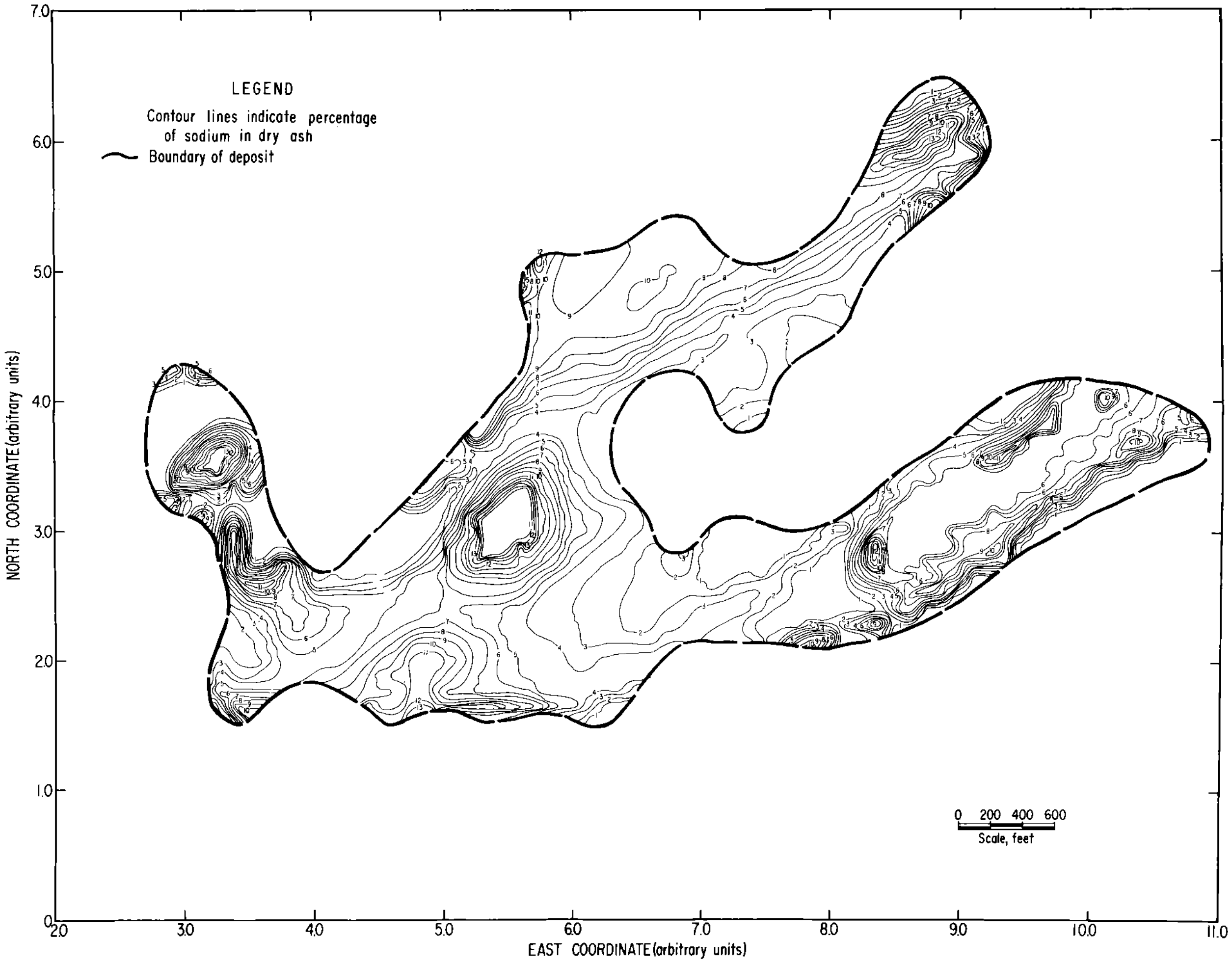

FIGURE 35. - Sodium Concentration in Lignite Deposits.

Other studies were made to develop statistical methods of predicting lignite chemical properties and potential low-temperature 500° C (932° F) carbonization yields.[32]

MHD Generation of Electricity

Power generation efficiencies of conventional steam-turbine generators might be increased considerably above the present plateau of 40 percent by coupling such units with a magnetohydrodynamic (MHD) generator. Reduction in sulfur oxide output and a substantial decrease in thermal pollution are other possible advantages of such a combination.

In MHD, electricity is generated by rapidly passing a hot ionized gas containing a so-called seed material across a magnetic field. Potassium carbonate has been under investigation as seed, with emphasis placed on the ability of the carbonate to remove sulfur dioxide from the combustion gases. Experiments were conducted in 1969 in which carbonate in suitable amounts for MHD operation removed 99 percent of the sulfur dioxide from the flue gas. In other work, a computer solution for complex chemical systems was devised to elucidate reactions in the high-temperature combustion of coal associated with MHD power generation.[33]

Regeneration of the sulfur-laden seed continued to be studied.[34] Regeneration tests with spent seed-slag mixtures indicated that 90 percent of the sulfur in H_2-H_2O and H_2-CO_2 systems could be removed. Thus, recycling this material to the process could produce an essentially sulfur-free gaseous effluent.

The high temperatures necessary in MHD produce high nitric-oxide levels. Nitric oxide output might be decreased and the conductivity of the gases increased by operating the combustor with 90 to 95 percent of the theoretically proper amount of air and adding the additional air downstream.[35] Tests are presently underway to investigate this.

Three sets of experiments were conducted with the MHD vortex generator. The major objective of these experiments is to study the vortex MHD principle;

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[32] Gomez, Manuel, and William L. Meyer. Application of Statistical Response Surface Fitting in Predicting Lignite Properties. BuMines Rept. of Inv. 7237, 1969, 64 pp.

[33] Feldmann, H. F., W. H. Simons, and D. Bienstock. Calculating Equilibrium Compositions of Multiconstituent, Multiphase, Chemical Reacting Systems. BuMines Rept. of Inv. 7257, 1969, 22 pp.

[34] Feldmann, H. F., W. H. Simons, J. J. Gallagher, and D. Bienstock. Kinetics of Recovering Sulfur From the Spent Seed in an MHD Power Plant. Preprint, Div. Fuel Chem., Am. Chem. Soc., Joint Symp. Fuel and Anal. Chem., Minneapolis, Minn., Apr. 13-18, 1969, v. 13, No. 2, pp. 13-26.

[35] Feldmann, H. F., W. H. Simons, and D. Bienstock. The Effect of Air/Fuel Level in the MHD Generator on the Operation of an Open-Cycle MHD-Topped Power Plant. 10th Symp. Eng. Aspects MHD, Mass. Inst. Technol., Cambridge, Mass., Mar. 26-28, 1969, pp. 142-144.

however, various materials are also being evaluated for use in the extremely high-temperature environment necessary for MHD generation of electricity. Modifications were begun in preparation for a fourth set of experiments. In the vortex generator, the flame from a cyclone burner follows a rotary path through a solenoidal magnetic field, resulting in migration of free electrons either to the zirconia shell of the chamber or to a coaxial center electrode, depending on the direction of the magnetic field. Because zirconia is a good conductor of electricity at high temperature, electrons flow through an externally connected circuit to generate electric power.

SECTION 7. - COKE, CHAR, AND CHEMICALS FROM COAL CARBONIZATION

Blending for Coke Manufacture

Blending relatively small proportions of low- and medium-volatile coals with high-volatile coals is standard practice at coke plants because individual coals do not have all the properties required to make satisfactory metallurgical coke. Blending coals of low- and medium-volatile matter content are added, mainly to improve coke strength. In the Western United States, the low- and medium-volatile coals must be shipped long distances, hence coke production costs could be reduced if a suitable substitute could be found closer at hand. In a search of a substitute, char and coke breeze were used instead of low- and medium-volatile coals in blends with Lower Sunnyside coal from Utah.[36] The latter is presently being used by two major Western coke plants as a large proportion of their coking blends. The optimum blend contained 6 percent char (18.7 percent V.M.) and 6 percent coke breeze, and yielded coke with a 1-inch tumbler index (stability factor) of 43. This is slightly higher than the index of coke produced by plants in the same area. Addition of 10 percent low-volatile Pocahontas No. 3 bituminous or medium-volatile Sewell bituminous coals from West Virginia produced an even better coke.

Another investigation also seeks to determine the feasibility of substituting coal char for at least part of the relatively expensive blending coals now making up 10 to 20 percent of all Western coking blends. Reportedly, char substitution has been technically successful with some coals, but the principles leading to successful substitution are not well understood. In this investigation, volatile matter content of the char and concentration of char in the blend are major variables.

Two Western coking coals have been studied so far, both are high-volatile A bituminous in rank: Sunnyside, Carbon County, Utah; and Allen, Las Animas County, Colo. Chars of selected volatile matter contents were produced by entrainment carbonization, blended in various proportions with coal, coked in an experimental oven, and tested for quality. Addition of any of the several chars (including two lignite chars) to the coals studied, in concentrations up to 15 percent, produced markedly better coke than was obtained from unblended coals. Also, selected chars of certain volatile matter content, without the

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[36] Kovalik, M. J. D. E. Wolfson, F. Fischler, and L. Mafrica. Use of Antifissurants in Making Better Coke From Sunnyside Coal From Utah. BuMines Rept. of Inv. 7235, 1969, 15 pp.

use of blending coals, produced coke that meets industrial coke standards for Western blast furnaces. Higher quality coke would probably require some use of blending coals.

Carbonization Processes

Char

Conversion of coal into low-sulfur powerplant fuel (char) and a tar amenable to hydrogenation is the ultimate objective of research on the rapid (flash) carbonization of bituminous coal. Of primary interest is a continuous process to carbonize high-volatile bituminous coals at high throughputs. Work with a 4-inch-diameter by 1-foot-long carbonizer established the processing conditions that give optimum char yields and quality for both internally or externally heated carbonizers.[37] Optimum char quality (a 15-percent V.M. product) at the highest processing rate (750 grams per hour) was achieved by carbonizing at 1,900° F with the heat applied internally. Under these conditions, char was produced at a rate equivalent to almost 1,100 pounds per ton of coal carbonized. In work to establish maximum coal throughputs, almost 600 pounds of coal per square foot of carbonizer cross-sectional area was processed per hour (fig. 36). At coal rates up to about 500 pounds per square foot per hour, total heat input significantly influenced the amount of volatile matter in the product char; above 600 pounds per square foot per hour, neither heat input nor coal rate had much effect. Uniform-quality chars containing 10 to 25 percent volatile matter have been produced in quantities large enough for product testing.

Carbonization Product Upgrading

High-splint coal from Kentucky was carbonized in a fluid-bed carbonizer (using, alternately, steam or recycle gas as the fluidizing medium) and the tar vapors cracked at 600°, 700°, and 800° C (1,112°, 1,292°, and 1,472° F) in a fluid-bed of high-temperature coke to determine the effect on the decomposition products.[38] Upgrading the products to ones having greater value is the ultimate aim. Yields of tar, pitch, acids, and bases were higher when recycle gas served as the fluidizing medium.

In another investigation,[39] increasing the free space above the charge in coal carbonization from 1 inch to 9 inches decreased the tar yields and

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[37] Belt, Robert J., John J. S. Sebastian, and John S. Wilson. Continuous Rapid Carbonization of Powdered Coal by Entrainment: Response Surface Analysis of Data. Preprints, Div. Fuel Chem., Am. Chem. Soc., 157th Nat. Meeting, Minneapolis, Minn., Apr. 13-18, 1969, v. 13, No. 2, pp. 110-121.

[38] Ortuglio, C., J. G. Walter, and D. E. Wolfson. Effect of Increasing Free Space Above the Charge on the Composition and Yield of the Volatile Products From Coal Carbonization. Prepints, Div. Fuel Chem., Am. Chem. Soc., New York, Sept. 7-12, 1969, v. 13, No. 4, pp. 71-79.

[39] Sanner, W. S., B. W. Naugle, and D. E. Wolfson. Fluidized-Bed Low-Temperature Carbonization of Bituminous Coal and Thermal Cracking of the Tar Vapors. BuMines Rept. of Inv. 7322, 1969, 19 pp.

increased the gas and light oil yields. Anthracene and naphthalene yields were higher, as were the quinoline- and benzene-insoluble fractions of both the tar and pitch, although the carbon content of the tars and pitches increased only slightly. Since coke is the most valuable product of coal carbonization, it would not be economically feasible to decrease the capacity of commercial ovens by increasing the free space above the charge. Based on the information obtained, investigation was begun of upgrading the volatile materials in a separate cracking unit outside of the coking furnace.

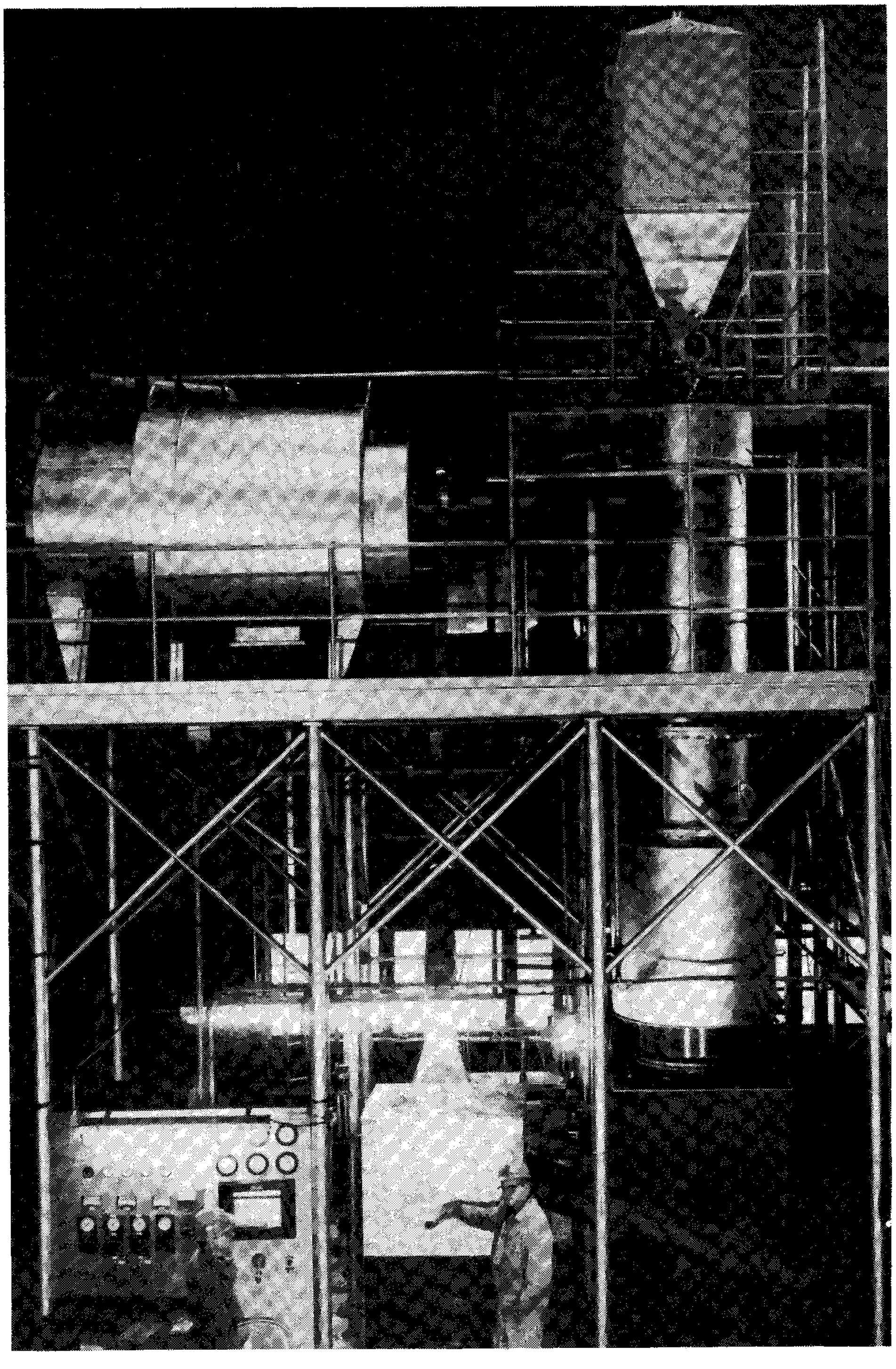
FIGURE 36. - Carbonizing Coal Into Low-Sulfur Char for Powerplant Fuel.

Charcoal From Waste Wood

A report[40] was published giving results of an exploratory study of wood waste carbonization. Entrainment carbonization techniques originally developed for coal were found readily applicable to crushed waste

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[40] Boley, Charles C., and W. S. Landers. Entrainment Drying and Carbonization of Wood Waste. BuMines Rept. of Inv. 7282, 1969, 15 pp.

from typical hard and soft woods. Wood wastes accumulate in vast amounts in the lumber and wood products industries.

Activated Carbon

Production of activated carbon by a fluid-bed reactor was studied under a Bureau of Mines-University of North Dakota fellowship program.[41] Temperature and residence time were both significant with respect to activation, and the activated carbon produced was equal to or better than commercially available activated carbon, as measured by iodine and color adsorption tests. The best product was obtained at an activation temperature of 1,750° F and a residence time of 24 minutes.

Low-Temperature Carbonization

Process Development

Extensive research was conducted on the effect of changing carbonization conditions on the composition of low-temperature coal tars.[42] The tars were produced in an inert atmosphere at 500° C (932° F) from seven different coals representing the major coal ranks. Analyses were made for the amounts of about 100 compounds and several classes of compounds, in each tar and its corresponding light oil, in terms of the coal as it would be charged in a commercial process. Enough information was obtained on compounds of commercial interest to permit economic evaluation and comparison with tars and light oils from coals of various rank carbonized under the same conditions.

Product Upgrading

A program to determine the technical feasibility of producing chemicals from low-temperature lignite tar was completed during 1969. The final phase dealt with the preparation of carbon black from the thermal decomposition of low-temperature lignite pitch. Carbon black yields increased from 16 weight-percent of the pitch fed at 1,800° F to 37 weight-percent at 2,500° F, which is equivalent to 40 pounds of black per ton of lignite carbonized. Electron micrographs showed that the particles were spherical in shape, ranged from 200 to 3,000 Angstroms in diameter, and had a tendency toward arrangement into chainlike structures.[43]

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[41] McNally, John R. Activated Carbon From North Dakota Lignite. M.S. Thesis, University of North Dakota, Grand Forks, N. Dak., 1969, 55 pp. (Available from University of North Dakota library.)

[42] Comberiati, Joseph R., Clarence Karr, Jr., Edward E. Childers, Jesse O. Mapstone, Jr., and Patricia A. Estep. Effect of Coal Rank on Composition of Low-Temperature Tar From a Bench-Scale Fixed-Bed Carbonizer. Preprints, 158th Ann. Meeting, Div. Fuel Chem., Am. Chem. Soc., New York, Sept. 7-14, 1969, v. 13, No. 4, pp. 160-178.

[43] Rice, Richard L., Robert E. Lynch, and John S. Berber. Carbon Black Preparation From Low-Temperature Lignite Pitch. Symposium on Chemicals From Coal. Preprints, Div. Fuel Chem., Am. Chem. Soc., New York, Sept. 7-12, 1969, v. 13, No. 4, 1969, pp. 199-206.

Carbon electrodes were prepared from tar produced by low-temperature carbonization of lignite. Coke aggregate for the electrodes was produced by delayed coking of pitch obtained by distilling low-temperature lignite tar; the pitch served as electrode binder. Electrodes prepared from these materials compared favorably with commercial high-purity electrodes.[44]

Thermal cracking of pitch from the carbonization of Texas lignite at low temperature has been under investigation as a means of producing aggregate and a binder that could be used to fabricate carbon metallurgical electrodes. A previous report on this subject, covering preliminary tests on the thermal cracking of lignite pitch at 1,450° F in a 2-1/2-inch-diameter reactor, showed that a variety of products could be obtained. In 1969, a 4-inch diameter reactor was used to evaluate oil, coke, and gas quality and gas yields as a function of pitch feed rate and temperature between 1,200° and 1,450° F.[45]

Coking Coal Reserve Surveys

Alaska

Coking studies have been made on samples of several interesting coal deposits in Alaska in cooperation with the Alaska Office of Mineral Resources. A report summarizes the latest results.[46] Previously reported information that a 20-foot bed in the Kukpowruk River area contains a potentially satisfactory base-coking coal was confirmed, and single samples from the Kokolik River and Cape Beaufort areas were found to have definite coking characteristics.

Appalachia

Carbonizing properties were determined for the principal bituminous coalbeds in Nicholas, Randolph, and Webster Counties in West Virginia.[47] Carbonization tests were made by the BM-AGA method at 900° C (1,652° F) because the quality of products thus obtained approximates the quality from industrial carbonization. Thirty-three samples were carbonized, representing high-volatile A coals in Middle Kittanning, Lower Kittanning, Coalburg, Winifrede, Peerless, Eagle, Lower Eagle, and Sewell beds in Nicholas County; Lower

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[44] Berber, John S., and Richard L. Rice. Preparation of Carbon Metallurgical Electrodes From Low-Temperature Lignite Coke and Lignite Pitch Binder. I&EC Product Research and Development, v. 8, June 1969, p. 188.

[45] Rice, Richard L., Delmar R. Fortney, and John S. Berber. Thermal Cracking of Low-Temperature Lignite Pitch. Part II. Preprints, Div. Fuel Chem., Am. Chem. Soc., Minneapolis, Minn., Apr. 13-18, 1969, v. 13, No. 1, 1969, pp. 122-129.

[46] Warfield, R. S., and Charles C. Boley. Sampling and Coking Studies of Several Coalbeds in the Kokolik River, Kukpowruk River, and Cape Beaufort Areas of Arctic Northwestern Alaska. BuMines Rept. of Inv. 7321, 1969, 58 pp.

[47] Wolfson, D. E., and C. Ortuglio. Carbonizing Properties of Coals From Nicholas, Randolph, and Webster Counties, W. Va. BuMines Rept. of Inv. 7236, 1969, 17 pp.

Kittanning, Peerless and Sewell beds in Randolph County; and Peerless and Sewell beds in Webster County; also, medium-volatile Sewell coals in Nicholas and Randolph Counties and Fire Creek bed in Webster County. Chemical composition of most of the coals investigated were excellent for metallurgical coking. Except for four samples of Lower Kittanning coal in Nicholas and Randolph Counties, the ash and sulfur-content ranges were 1.8 to 9.0 percent and 0.4 to 1.1 percent, respectively. Peerless coal in the three counties contained 1.8 to 4.7 percent ash and 0.7 to 1.1 percent sulfur; eight of the 11 samples of Sewell coal contained 2.5 to 4.3 percent ash, and 10 of them contained 0.5 to 0.9 percent sulfur. All coke strengths were within the normal range of Appalachian coals of similar rank.

Continental United States

Appraisal of the extent and availability of metallurgical quality coals in the United States was continued, on a limited basis, in conjunction with the survey of deposits of low-sulfur coals, with which many coking coals are synonymous. Continued availability of these coking coals is essential to meet the predicted greater demands by the coke and steel industries and to maintain a high level of coal exports, with resulting credits to our international balance of payments. Air pollution regulations also make it essential that sources of high-quality coals be charted.

SECTION 8. - FUELS AND CHEMICALS FROM COAL BY SYNTHESIS PROCESSES

Pipeline Gas

Gasification Process Development

Fluid-Bed Gasification

Work was continued toward producing high-Btu fuel gas by fluid-bed gasification of both weakly and strongly caking coals. Maintenance of a high methane concentration at the gasifier outlet and a high gasification rate is sought to significantly improve the overall process. Caking coals must be pretreated to make them noncaking before they can be gasified in a fluid bed, and significant carbonization must be effected in fluid-bed gasification to produce a methane-rich product gas. Pittsburgh seam bituminous coal has been successfully processed at 20 atmospheres in an integrated unit combining pretreatment and fluid-bed carbonization and gasification to yield a gas containing 15 to 20 percent methane.[48] To increase the methane content and coal throughput, tests were begun in 1969 with a new unit operating at a 40-atmosphere pressure. Gasification of highly caking Pittsburgh seam coal was only partially successful at 40 atmospheres: The coal seemed to resist decaking at conditions that were successful at 20 atmospheres. On the other hand, Illinois No. 6, a medium caking bituminous coal, was gasified at 40 atmospheres with no evidence of agglomeration at a throughput of 306 pounds per hour per square foot, carbon conversion of 41 percent, and methane in the

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[48] Forney, A. J., S. J. Gasior, and J. H. Field. Process for Gasifying Caking Coals. U.S. Pat. 3,463,623, Aug. 26, 1969.

product gas of 14 percent. Modification of the unit is being considered to improve the pretreatment section, increase carbon conversion, and eliminate clinker formation.

Exploratory studies were continued in 1969 on a method for making synthesis gas containing less than 15 percent nitrogen even though air instead of oxygen is used to generate the gasification heat.[49] Successful substitution of air would appreciably reduce production costs. Oxygen for the gasification of coal can account for more than 20 percent of the total cost of making high-Btu gas from coal.

In the initial design investigated, gasification was effected in concentric tubes (6-inch tube inside an 8-inch tube) in which the products from steam-coal gasification passed up through the center tube and were heated by air-combustion products of coal passing between the tubes. The double tube arrangement was satisfactory for establishing the technical practicality of the method, but such a design is not adaptable to commercial use. Other designs are being investigated, including the multitube reactor shown in model form in figure 37. The single center tube is replaced by five smaller tubes.

In other fluid-bed gasification work, a pilot plant was virtually completed in 1969 for use in evaluating a novel process for coal gasification. In this gasifier, the coal combustion reactions that provide reaction heat are separated from the steam-carbon reactions that produce the synthesis gas (fig. 38). Separation of the combustion reactions from the steam-carbon reactions is designed to permit the use of air rather than oxygen and to produce a synthesis gas free of nitrogen. Inert particles of solid, referred to as grog, are circulated continuously between a fluid-bed coal combustor that heats the grog and a fluid-bed gasifier for which the hot grog supplies the reaction heat. Caking-type bituminous coals that are not suitable for some gasification processes can be processed without pretreatment. Product gas from such a system can be processed into hydrogen or gaseous fuel.

Hydrogasification

Hydrogasification of coal to produce pipeline gas is being investigated with a reactor in which coal dropping through the unit is hydrogenated, and the char produced is further hydrogenated. Control of temperature during the heat-evolving char hydrogenation proved to be a major problem and led to an alternate design involving only one process step. In this concept, hydrogen instead of a mixture of methane and hydrogen is fed with the coal. Coal conversions of up to 52 percent have been achieved in experiments (fig. 39) with raw high-volatile A bituminous coal from the Pittsburgh seam to produce a gas containing a maximum of 50-percent methane. Work was continued to raise both these values.

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[49] Forney, A. J., R. F. Kenny, and J. H. Field. Gasification of Coal in a Partitioned Fluid-Bed Reactor. Preprints, 65th Nat. Meeting of AIChE, Symp. on Chem. Processing of Coal, Cleveland, Ohio, May 4-7, 1969, Paper No. 23b, 13 pp.

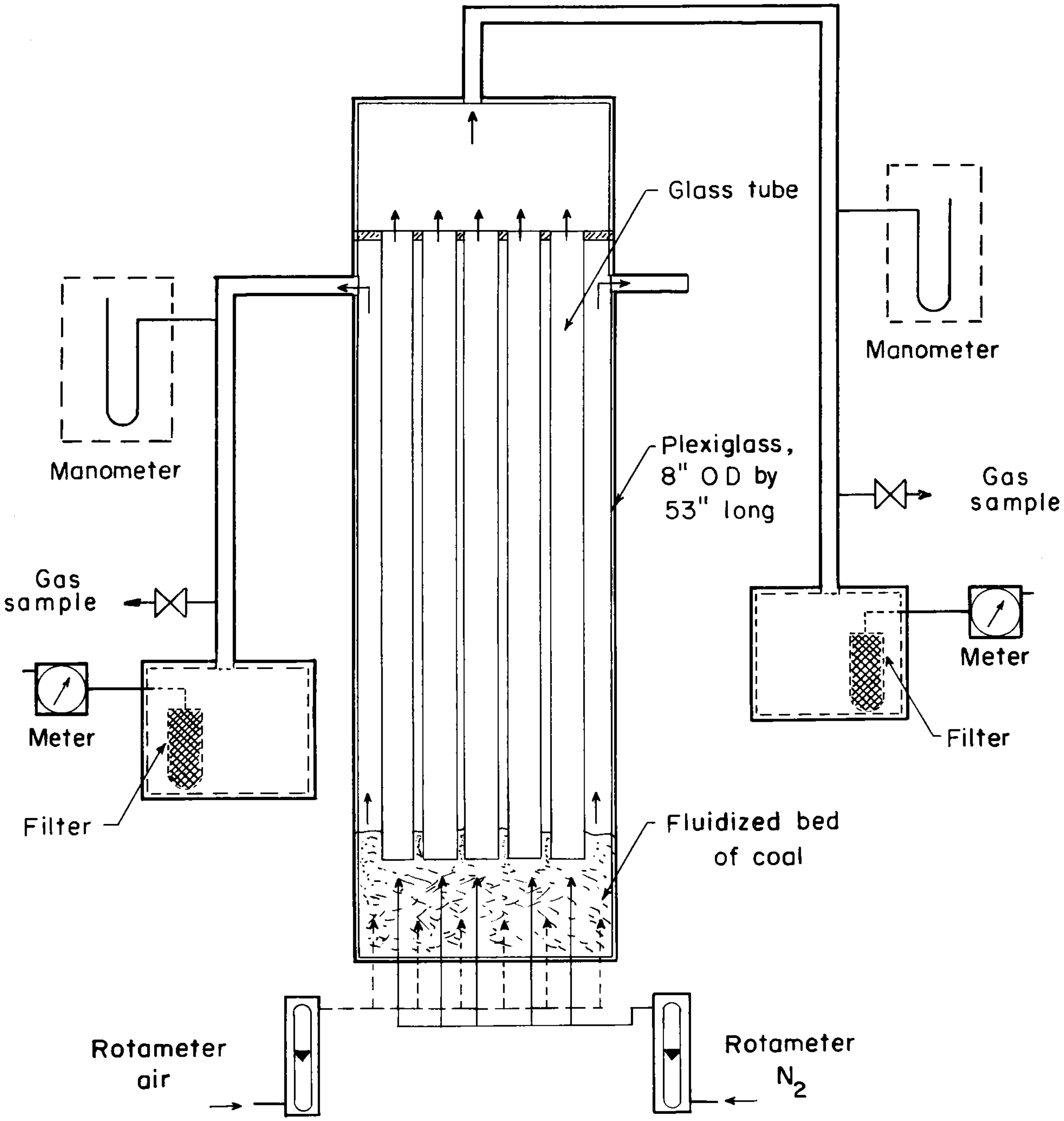

FIGURE 37. - Model of Multitube Reactor for Fluid-Bed Gasification of Coal.

Economic studies were also started on various modifications of the basic concept of one-step hydrogasification of coal coupled with additional processing to produce pipeline gas.

Fundamental Gasification Research

Kinetics of the steam-carbon reaction and related heat transfer mechanisms are being studied with the equipment shown in figure 40. Projected test limits of the system is 1,600° to 2,000° F and pressures to almost 100 psi. Reaction rate and heat transfer data on the steam-coal reaction are essential to the design and operation of coal gasifiers.

Gas Cleaning and Purification

Research was concluded on the electrostatic precipitation of solids from gases up to 1,200° F.[50] Also, a theory was developed to account for unwanted deposition of ash on the emission electrodes (wires) of industrial precipitators. Results of the precipitator research were correlated with findings from operations with a pilot-scale precipitator designed to clean hot gases (1,470° F) under oxidizing conditions.[51] A patent was awarded covering a process and apparatus for electrostatic separation of solid particles from high-temperature-streams of relatively low densities.[52]

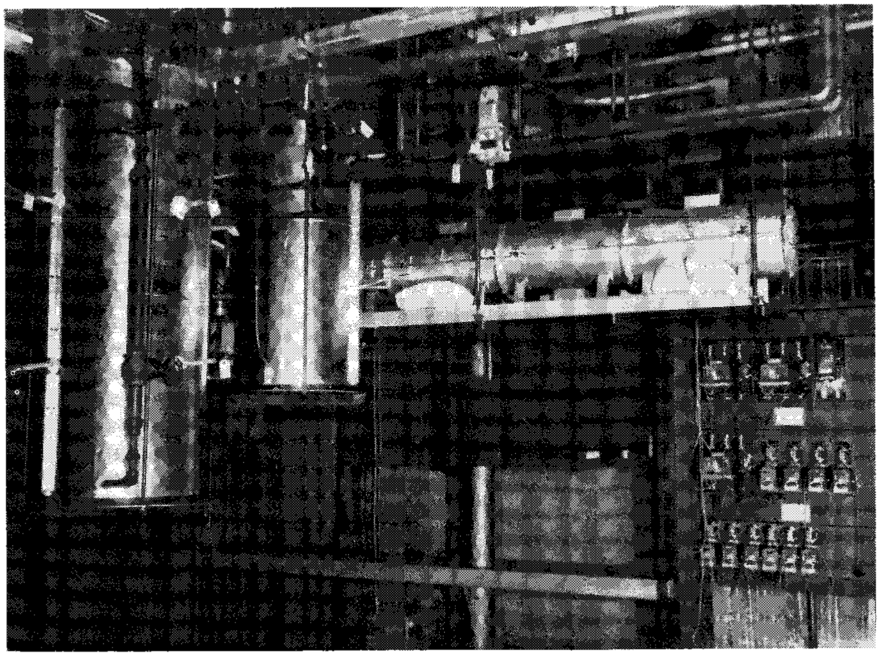

FIGURE 38. - Fluid-Bed Gasification Pilot Plant.

Gas Synthesis

Research was continued on the catalytic conversion of coal-derived synthesis gas into pipeline gas and hydrocarbons comprising gasoline. A bench-scale tube-wall reactor designed to effectively remove reaction heat to control the temperature of the catalyst has been demonstrated capable of producing pipeline-quality gas for extended periods. Because catalyst life has been considerably shorter in pilot-plant

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[50] Shale, C. C., and J. H. Holden. The Role of Wire Size in Negative Electrical Discharge at High Temperature. Trans. IEEE Industry and General Applications, v. IGA 5, No. 1, January-February 1969, pp. 34-39.

[51] Shale, C. C., and G. E. Fasching. Operating Characteristics of a High-Temperature Electrostatic Precipitator. BuMines Rept. of Inv. 7276, 1969, 19 pp.

[52] Shale, Correll C. Process for Electrostatic Precipitation. U.S. Pat. 3,482,374, Dec. 9, 1968.

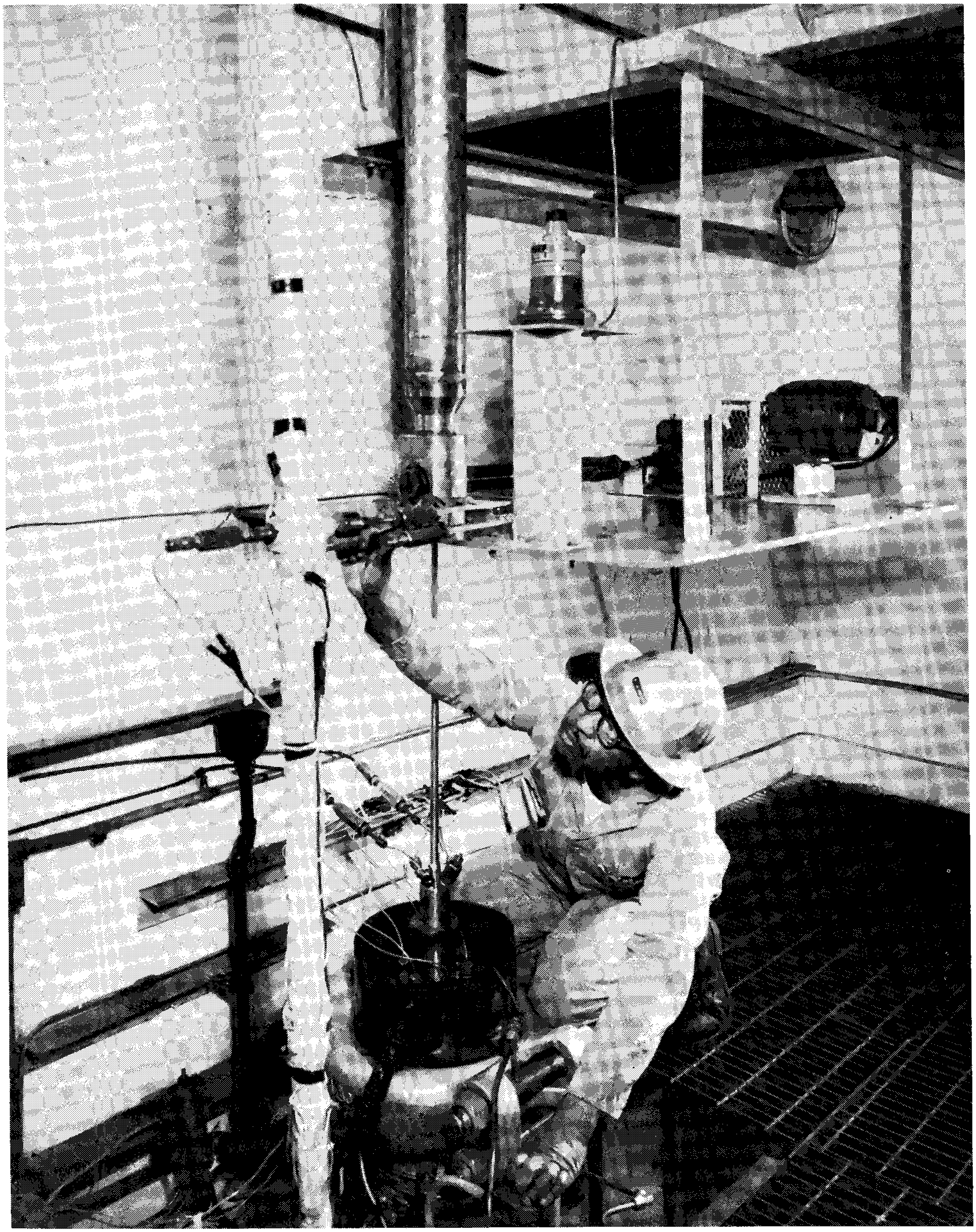

FIGURE 39. - Coal Hydrogasifier.

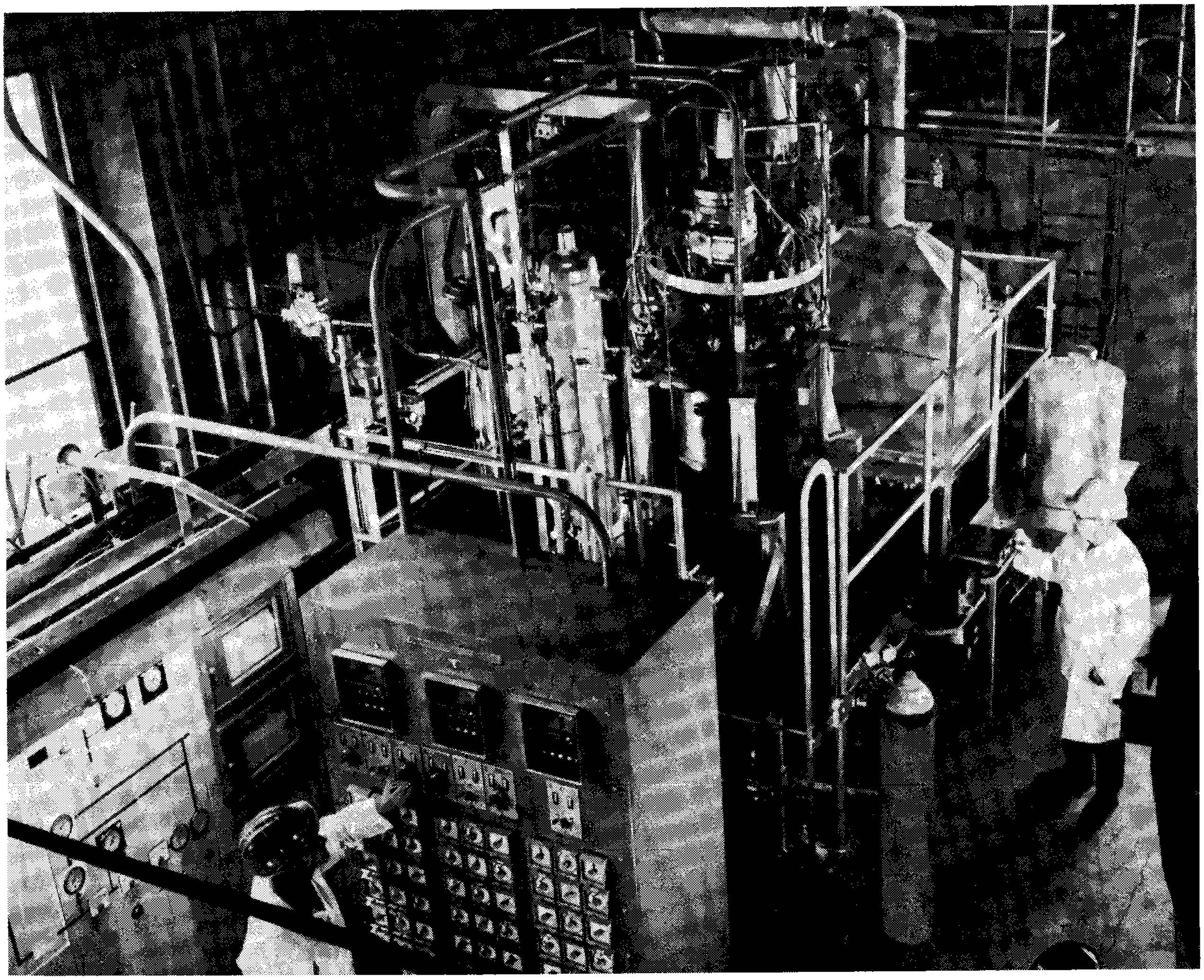

FIGURE 40. - Studying Reactions and Heat Transfer in Coal Gasification.

operations, bench-scale operations have been resumed to study reasons for this and to investigate methane production at pressures up to 1,000 psi. Various factors are being studied for their influence on catalyst life and performance.

Synthesis of gasoline-forming hydrocarbons was investigated further with a hot-gas recycle plant containing parallel metal plates coated with Alan-wood magnetite as catalyst. Alan-wood magnetite is a cheap iron ore concentrate, and for this application was impregnated with potassium carbonate (about one-tenth of 1 percent by weight of K_2O after reduction) to favor production of oil rather than gas. At a synthesis pressure of 400 psi, average catalyst temperature of 320° C (608° F), fresh gas composition of 1.4 parts H_2 to 1 part CO, space velocity of 1,000 standard cubic feet per cubic foot of catalyst volume per hour, and a cold-recycle- to fresh-gas ratio of about 2 to 1, conversion of CO + H_2 was 93 percent; the gasoline fraction contained about 50 percent of the hydrocarbon products. Considering the rather high space velocity, gasoline product yield was good.

Coal Pretreatment

Coal pretreatment studies were continued in an effort to produce a noncaking char suitable for various gasification processes. Caking coals, found mostly in Eastern United States, become soft and sticky at 300°-400° C (572°-752° F) and cannot be processed in fixed-bed and fluid-bed gasifiers. Methods have been developed by the Bureau of Mines for decaking up to 1-inch-size coal in a fixed-bed pretreater and up to 1/4-inch coal in a free-fall system. Since the free-fall method is more rapid, tests were continued in the 4-inch-diameter by 25-foot-long free-fall pretreater to find out if 3/8-inch Pittsburgh seam bituminous coal and larger can be decaked into a noncaking char suitable for processing in commercial Lurgi gasifiers. The tests were performed at 50 to 350 psig, wall temperatures of 560° to 650° C (1,040°-1,200° F), instantaneous coal temperature of 750° C (1,382° F), and with gas-to-coal ratios up to 114 cubic feet of steam per pound plus up to 4.0 cubic feet of oxygen per pound of coal. Such treatment produced little or no change in the caking quality of 1/4- to 1/2-inch coal. Coal particles (measuring 0 to 1/4 inch) that broke off from the larger pretreated particles usually appeared noncaking; the free swelling indices were of 2 or less.

Producer Gas

Producer gas, a fuel gas with relatively low calorific value, is made by passing steam and oxygen continuously through a deep bed of incandescent coal or coke, with the oxygen supplied either as air or pure oxygen. Producer gas production reached a peak in the United States in about 1925 when more than 12,000 producers were in operation. Shortly thereafter, producers went into a decline and producer gas was almost entirely supplanted by natural gas. Contributing to the decline of producers was their limitation to noncaking coals, a factor that restricted fuel availability and boosted process costs. Most of the coal deposits east of the Mississippi River are caking types that, when heated, agglomerate into a mass that is impermeable to gas flow.

A fixed-bed gas producer (fig. 41) designed for 300 psi and coal throughputs to 150 pounds per square foot per hour was built and has been operated in several series of tests. One of the main objectives of these tests has been to successfully gasify all grades of coal by providing mechanical stirring of the fuel bed, a feature that distinguishes the experimental producer from commercial fixed-bed producers. Promising results have been obtained thus far in short operating periods using strongly caking bituminous coals from Ohio and West Virginia. Coal throughputs of 140 pounds per square foot per hour have been attained at 80 psi; gas generation rates have approached 100,000 standard cubic feet per hour. Typical composition of the gas is as follows:

	Percent
Carbon monoxide.....	21
Hydrogen............	15
Carbon dioxide......	7
Methane.............	2
Nitrogen............	55

Gas of this quality would be satisfactory for firing industrial furnaces and kilns or, after tar and dust removal, driving a gas turbine.

Iron oxide (Fe_2O_3) will remove hydrogen sulfide from producer gas at temperatures in the 1,000° to 1,500° F range if the oxide is incorporated into a semifused porous matrix of other metallic oxides to prevent absorbent loss via dust formation. Bench-scale tests were made to determine the absorbing characteristics of a sintered mixture of Fe_2O_3 and fly ash and sintered red mud residues from aluminum refining. In the red mud experiments, sulfur capacities ranged as high as 53 percent and increased generally in proportion to the iron oxide content of the muds. Spent absorbents in both cases were regenerated to an essentially fresh condition by passing air through them at temperatures of 1,000° to 1,200° F. During regeneration, sulfur dioxide was evolved in high enough concentrations to permit recovery as sulfuric acid.[53]

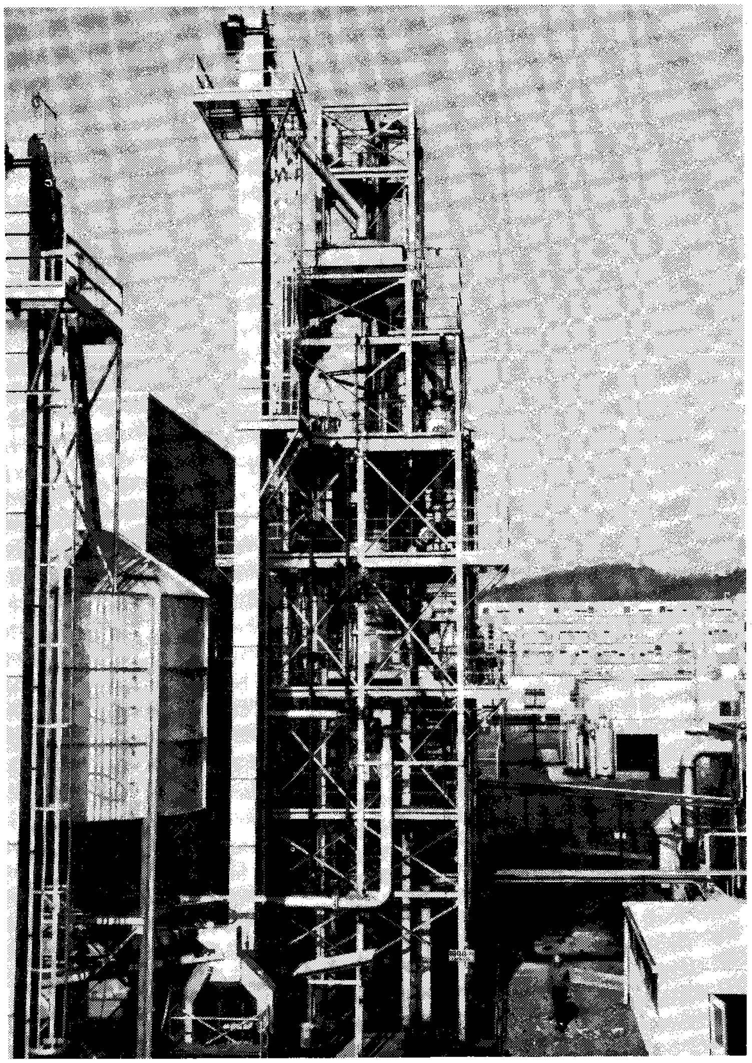

FIGURE 41. - Experimental Gas Producer for Caking-Type Bituminous Coals.

Liquid Fuels and Chemicals (Hydrogenation)

Process Development

Petroleum-processing catalysts are being evaluated for their effectiveness in hydrogenating coal tars and tar-coal mixtures into low-boiling oils. In experiments with high-temperature coal tar at 4,000 psi, 800° F, and feed rate of 5 pounds per hour, silica-promoted cobalt molybdate proved superior to non-silicated cobalt molybdate, nickel molybdate, sulfided nickel molybdate, and sulfided nickel tungsten catalysts. With this catalyst, a liquid product of constant composition (fig. 42) was produced continuously for 170 hours

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[53] Shultz, Forrest G., and John S. Berber. Hydrogen Sulfide Removal From Hot Producer Gas With Sintered Absorbents. General Papers. Preprint, Div. Fuel Chem., Am. Chem. Soc., New York, Sept. 7-12, 1969, v. 13, No. 4, 1969, pp. 30-36.

with no apparent deactivation of the catalyst. Benzene insolubles and asphaltenes in the liquid product were 5 percent and 6 weight-percent, respectively, compared with 14 percent and 45 weight-percent in the coal tar. Liquid yield, based on tar, was 94 percent; yields of oils boiling under 390°,

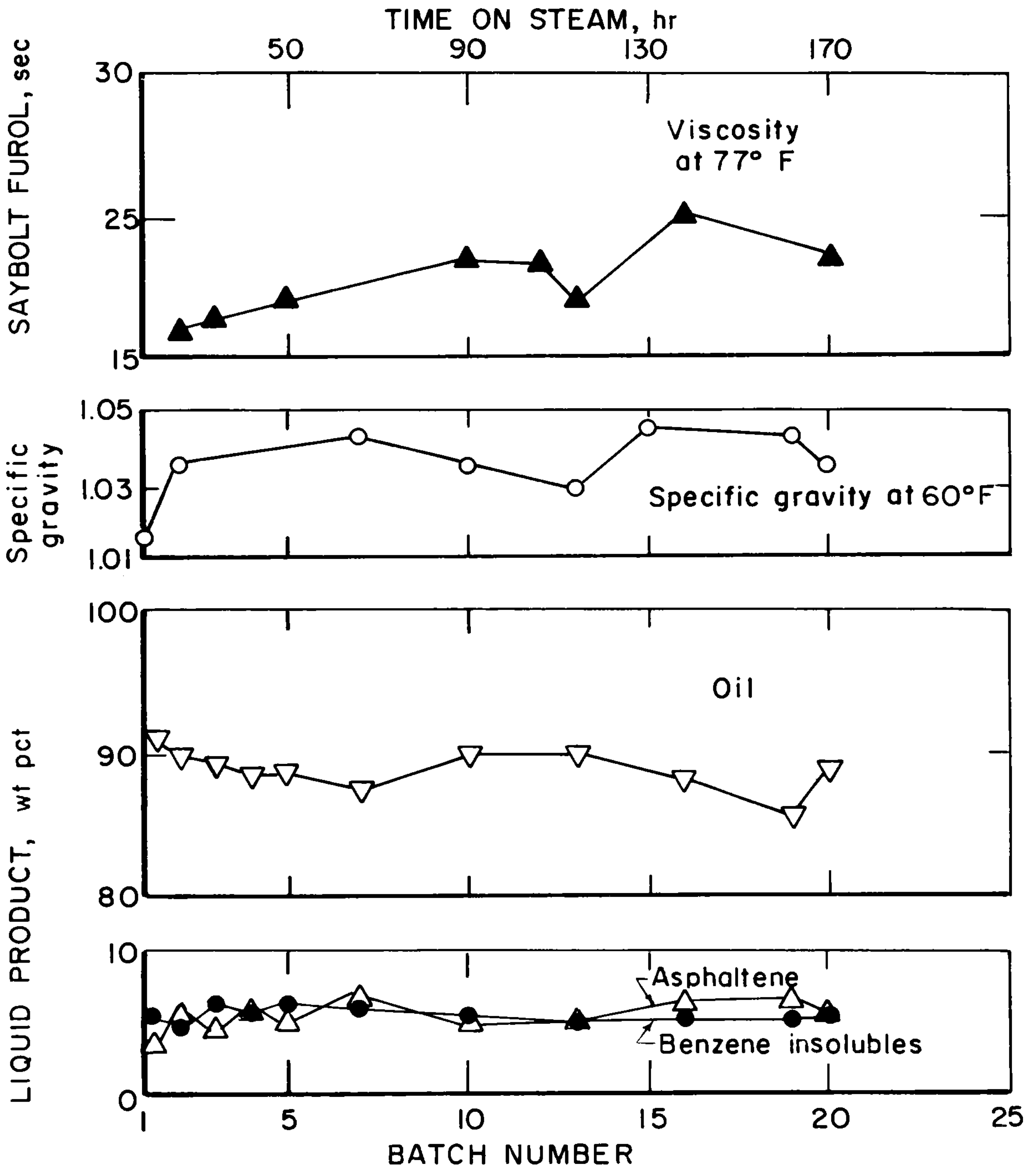

FIGURE 42. - Chart Showing Properties of Liquids Produced by Fixed-Bed Hydrogenation of Coal Tar With Silica-Promoted Cobalt Molybdate.

620°, and 670° F were 7, 40, and 45 percent, respectively. Work was continued with silica-promoted cobalt molybdate to establish the operating conditions for optimum conversion of coal to liquids.

Investigations were also initiated to determine the physical and chemical changes (fig. 43) in the catalyst resulting from use in hydrogenation. These changes may be important because regeneration of the spent catalyst would be essential in commercial applications.

Also being studied is the rate of conversion of coal to liquid fuels and the factors that influence these rates (fig. 44).

Coal liquefaction knowledge is also being extended by two studies underway on the hydrogenation of tar to low-boiling liquid hydrocarbons. A major objective of one of these studies is to determine the effect of thermal hydrogenation of tar residues using, as a hydrogen source, the so-called hydrogen donor diluents (HDD). With tetralin as HDD, hydrogen was transferred to a +1,000° F coke oven tar residue to produce an 11 weight-percent yield of gasoline. With a more reactive +800° F residue, the gasoline yield was doubled to 22 weight-percent. In this reaction, tetralin is dehydrogenated to naphthalene.

The objective of the other study is to determine the effect of catalytic hydrogenation of tars as they are evolved in the low-temperature carbonization of various coals in a hydrogen atmosphere. Such hydrocarbonization tests were made on Pittsburgh seam bituminous coal in which tar vapor, or both liquid tar and tar vapor, were passed through a catalyst bed at 900° to 1,000° F, the same temperature as the bed of coal. Typical yields of gasoline-rich product oil were 4 weight-percent from tar vapor only and 6 weight-percent from both liquid tar and tar vapor. However, when nitrogen was used instead of hydrogen, only 2.5 weight-percent was produced.

Liquid fuels production by the reaction of coal with carbon monoxide and water was subject to initial research effort in 1969 via the start of design and construction of a small continuous flow unit to evaluate this novel technique. Design operating conditions of this unit are 5,000 psi, 660° to 930° F, and a feed rate of 4 pounds per hour of coal-oil slurry.

Hydrogenation Catalysts

Experiments are underway to develop new and specific catalysts for liquefaction of coal by hydrogenolysis at moderate hydrogen pressures (2,000 to 2,500 psi). Of major interest is the development of catalysts that will increase coal conversion rates and yields of specific products. Attention is being directed particularly to catalysts that will disproportionate (rearrange molecules) asphaltenes, which are high molecular-weight intermediates formed when coal is liquefied. Cracking of asphaltene at 750° to 850° F, plus addition of hydrogen, forms gaseous hydrocarbons and lower molecular-weight compounds ranging from heavy fuel oil to gasoline. Asphaltenes are difficult to disproportionate, and the process proceeds quite slowly. Emphasized in 1969 was the development of catalyst from modified forms of silica-alumina

FIGURE 43. - Studying Changes in Catalysts Resulting From Use in Coal Tar Hydrogenation.

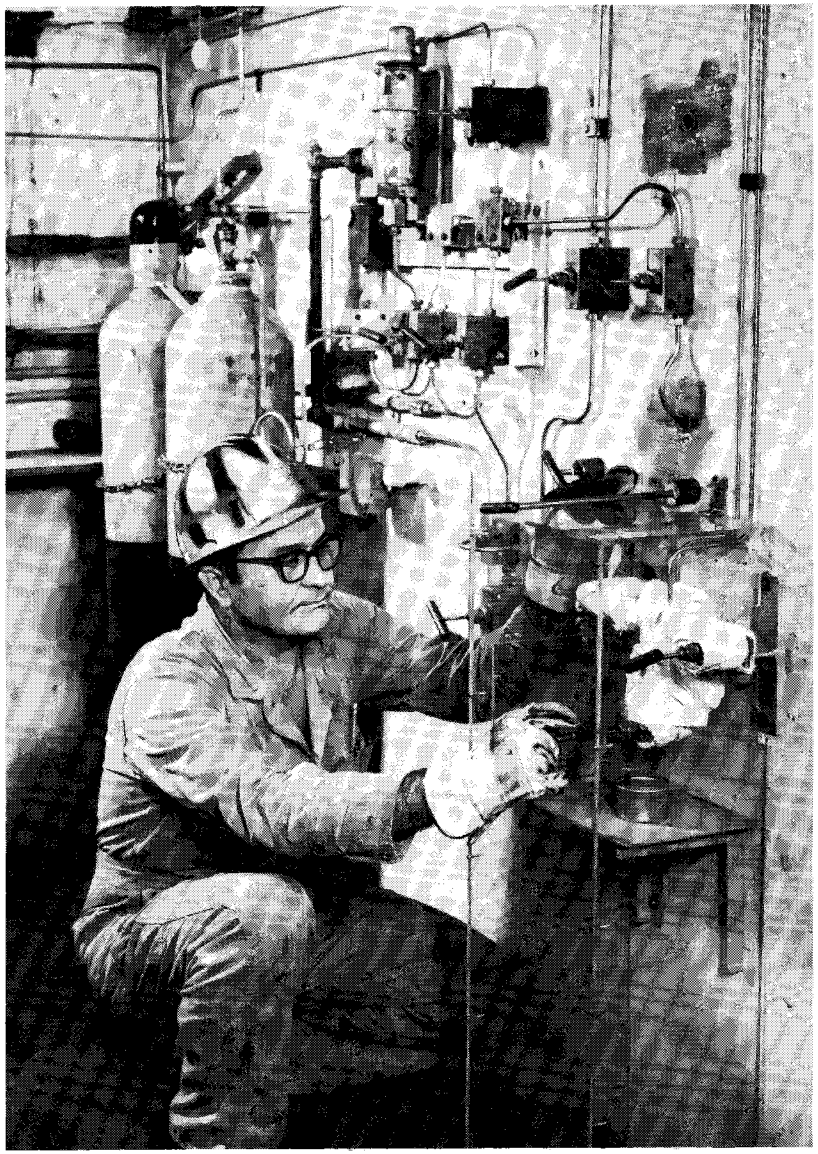

FIGURE 44. - Sampling Liquids Produced by Hydrogenation of Coal.

molecular sieves (synthetic zeolites) that contain sodium atoms in their crystal structure. These zeolites are being altered by replacing 60 to 65 percent of the sodium with metals such as molybdenum, cobalt, or tin, which are known to have good activity for hydrogenation. Thus a superior catalyst may result by combining the cracking property of silica-alumina with the hydrogen-adding property of certain metals.

Studies of various materials as catalysts for coal hydrogenation were continued. To date, more than 180 materials have been tested, of which more than 80 have shown catalytic activity for oil production. Halogen compounds of zinc, copper, iron, tin, nickel, mercury, and cadmium were very effective catalysts for the production of low-boiling oil in high yields. Commercial applications of the halide catalysts would necessitate developing efficient and low cost methods for their recovery and regeneration. Nonhalide catalysts generally produced oils consisting mainly of high-molecular weight nondistillable components.

Because the high-boiling oils produced in a continuous coal hydrogenation system would probably be used as a vehicle for injecting coal into the reactor, the effect of vehicle oil on the activities of about 40 coal hydrogenation catalysts was investigated. Activities of most of the catalysts were the same in the presence of vehicle oil as they were without the oil. The activities of cobalt molybdate and nickel molybdate were enhanced by the oil; copper bromide and zinc iodide suffered significant decrease in activity.

The primary product of coal hydrogenation using a conventional catalyst is asphaltene--a benzene-soluble and pentane-insoluble material. Hydrogenation of asphaltene to yield distillable oil is difficult and is the slow step in converting coal to oil. In experiments with a coal-derived asphaltene and 10 of the most active halide catalysts, hydrogenation of the asphaltene occurred quite readily with all of the catalysts. These results indicated that, in the conversion of coal to oil, the sequence of reactions is the same with high concentrations of halide catalysts as it is with conventional catalysts used in low concentrations.[54] The data also showed that the halides were selective catalysts for asphaltene hydrogenation.

Asphaltenes produced in the catalytic hydrogenation of coal were investigated because limited data is available concerning these intermediate products in the conversion of coal to oil. With MoO_3 as catalyst, an asphaltene was produced that had a higher molecular weight, was less volatile, and contained fewer heterocyclic ring structures than was produced without the catalyst. Asphaltenes produced with six other catalysts showed similar though much less pronounced differences relative to product from uncatalyzed hydrogenation.

Work has started on the preparation of a homogeneous catalyst held in a molecular sieve. A catalyst for the oxo reaction, made in this way,

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[54] Kawa, Walter, Sam Friedman, and R. W. Hiteshue. Selectivity of Coal Hydrogenation Catalysts. Preprints, AIChE, 64th Nat. Meeting, New Orleans, La., March 1969, Preprint 42A, 11 pp.

functioned well. Further application to the hydrogenation of coal will be pursued in 1970.

Phthalic Acids From Coal

Production from coal of phthalic acids, a group of chemicals with a potential market in the resins, plastic, and synthetic fibers industries, is the objective of process development work continued in 1969. One process involves the catalytic partial decarboxylation of a mixture of "coal acids," which are the acid-soluble products formed by the oxidation of coal suspended in an alkali medium. Bench-scale studies of the oxidation phase have established conditions under which 40 percent of the carbon in the coal can be converted to coal acids. The variables included temperature, pressure, and concentration and rank of coal. A small supply of the polycarboxylic acids have been produced for subsequent evaluation in the decarboxylation phase of the study.

These coal acids are rich in benzenepolycarboxylic acids which, by selective decarboxylation, are converted into isophthalic and terephthalic acids. Both of the latter are valuable raw materials for polymer manufacture. In studies of the mechanism of the decarboxylation reaction, pyromellitic acid, the principal component of coal acids, was found to behave differently from any of the other benzenepolycarboxylic acids toward the cobalt carbonyl catalyst.[55] Pyromellitic acid is the only one of the acids that requires stoichiometric amounts of catalyst to undergo decarboxylation. This difficulty can be circumvented by converting the acid to its anhydride by sublimation. Sublimation also converts all of the other benzenepolycarboxylic acids found in a representative coal acid to their anhydrides and separates them from the unreactive humic acid portion of the product. As a result, it may be possible to eliminate several steps in the process for converting crude, oxidized coal to isophthalic and terephthalic acids. A stabilized form of cobalt carbonyl catalyst has been developed in which the carbonyl is formed in situ on an inert zeolite support. This catalyst has been found capable of isolation and reuse without further treatment to recover the cobalt.

Coal Solubilization

A new method was devised for introducing alkyl groups to convert coal into a benzene-soluble product. This alkylated coal is almost completely soluble in benzene, and its molecular weight (number average) was found to be from 2,800 to 3,000, the same range as the molecular weights of petroleum asphaltenes. This suggests that coal and petroleum asphaltenes differ in molecular structure, not molecular size.[56]

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[55] Friedman, S., S. R. Harris, and I. Wender. Partial Decarboxylation of Coal Aromatic Acids by Transition Metal Complexes. Preprints, Div. Fuel Chem., Am. Chem. Soc., v. 13, No. 4, September 1969, pp. 179-184.

[56] Markby, R. E., R. A. Friedel, S. Friedman, and H. W. Sternberg. Light-Induced Chemiluminescence in Derivatives of Coal and Petroleum. Preprints, Div. Fuel Chem., Am. Chem. Soc., September 1969, v. 13, No. 4, pp. 80-84.

Low-volatile B bituminous coal had been successfully solubilized previously by giving it a negative charge (converting it to an anion). More recently, coal of this rank was solubilized by giving it a positive charge. Up to 70 percent of a low-volatile bituminous coal was solubilized by treatment with methanesulfonic acid in nitrobenzene solution, and the insoluble residue retained more than 80 percent of the ash. Solubilization probably results from formation of a polyelectrolyte-type "coal cation" produced by protonation of aromatic structures. This work offers the prospect of a new method of solubilizing coal and removing the ash.

Aromatic hydrocarbons in coal tar represent a potential source of raw material for making plasticizers and polymers if a method can be devised to selectively reduce these hydrocarbons to compounds containing olefinic double bonds. Selective electrochemical reduction of aromatic hydrocarbons was achieved in ethanol-HMPA solution by proper control of current density, alcohol concentration, and temperature.[57]

Factors promoting increased solvation of ultrasonically irradiated coal-solvent slurries and of treated coals were investigated by spectral methods.[58] In the ultrasonic irradiation of coal-quinoline slurries, yields of solvated material were reduced by the presence of methyl derivatives of quinoline or water. Increasing the temperature from 36° to 80° C (97°-176° F) did not increase coal solvation.

SECTION 9. - NEW COAL PRODUCTS AND USES

Carbon Black

Carbon black made by partial thermal decomposition of finely ground coal by a process devised by the Bureau[59] was tested by several industrial companies and found comparable with thermal grades of black available commercially. Powdered coal transported by carrier gas down through an externally heated ceramic reactor tube is pyrolyzed into a char and a gas that contains the carbon black that is subsequently removed from the gas.[60] The product gas has been used successfully instead of nitrogen as the carrier gas. Also, recirculation of the byproduct char to the reactor increased carbon black yields (based on weight of coal fed) from 20 percent to about 25 percent and raised

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[57] Sternberg, H. W., R. E. Markby, I. Wender, and D. M. Mohilner. Reduction of the Benzene Ring and of the Olefinic Double Bond by Electrolytically Generated Electrons. J. Am. Chem. Soc., v. 91, No. 15, July 16, 1969, pp. 4191-4194.

[58] Kessler, T., R. Raymond, and A. G. Sharkey, Jr. Composition of Pyridine Extracts of Reduced and Untreated Coals as Determined by High-Resolution Mass Spectrometry. Fuel, v. 48, No. 2, April 1969, pp. 179-186.

[59] Johnson, G. E. Production of Carbon Black From Coal. U.S. Pat. 3,424,556, Jan. 28, 1969.

[60] Johnson, G. E., W. A. Decker, A. J. Forney, and J. H. Field. Carbon Black Produced by the Pyrolysis of Coal. Preprint, Div. Fuel Chem., Am. Chem. Soc., 158th Nat. Meeting, New York, Sept. 7-12, 1969, v. 13, No. 4, pp. 207-222.

product gas yields (about 80 percent hydrogen) to about 10 cubic feet per pound of coal.

Cost estimates indicate that carbon black can be made from coal by this process for slightly less than 2-1/4 cents per pound. Based on the current market price of thermal black of 6-1/4 cents per pound and a depreciation over a 20-year period, the rate of return on the investment is estimated as 23 percent.

Leonardite as Growth Regulator-Fertilizer

Final tests of potatoes grown in 1968 in leonardite-treated soils indicated that the treatment increased yields and had no detrimental effect on taste, sugar content, or chipping characteristics. A second large-scale planting was carried out in May 1969, in cooperation with the Horticulture Department of North Dakota State University (fig. 45). Some differences in plant size and pigmentation were observed at first, but this became less apparent as growth progressed. Growing conditions were ideal through early August 1969; however, the effects of drought and blight were evident in numerous areas. Hence, the somewhat better appearance of the test plot may indicate a beneficial effect from the leonardite. Preliminary yield data from the test plot, however, appear lower than for 1968. Firm conclusions are not yet possible.

Soybeans were planted in May 1969, but the results were not as good as those of the year before. Factors other than the presence or absence of leonardite in the soil appear to have been involved. Laboratory work was continued in an effort to establish the effect of leonardite on crop growth and quality.

Potential agricultural applications of lignite were reviewed in an article prepared on request for the Greater North Dakota Association.[61]

SECTION 10. - STRUCTURE OF COAL AND COAL DERIVATIVES

Coals, Cokes, and Carbons

Studies of atomic and molecular structures in coals, cokes, and carbons by X-ray scattering have been aided by improved methods of collecting diffracted intensity data and of correcting intensity distributions when experimental refinements are difficult. For example, a multichannel analyzer has been applied to accumulate statistically significant intensity data.[62] The intensities of X-rays diffracted by the specimen in different directions are measured at small angular steps, and the counts at each angle are accumulated in one of the 1,024 channels; the final totals are punched on paper tape for computer analysis. A complete pattern is recorded in about 15 minutes,

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[61] Freeman, Philip G., and Walter W. Fowkes. Lignite and Agriculture: A Marriage of Industries? North Dakotan, v. 44, No. 9, 1969, pp. 18-19.

[62] Berman, Martin, and Sabri Ergun. Discrete Averaging of X-Ray Diffraction Data Using a Multichannel Analyzer. Rev. Sci. Instr., v. 40, September 1969, pp. 1144-1145.

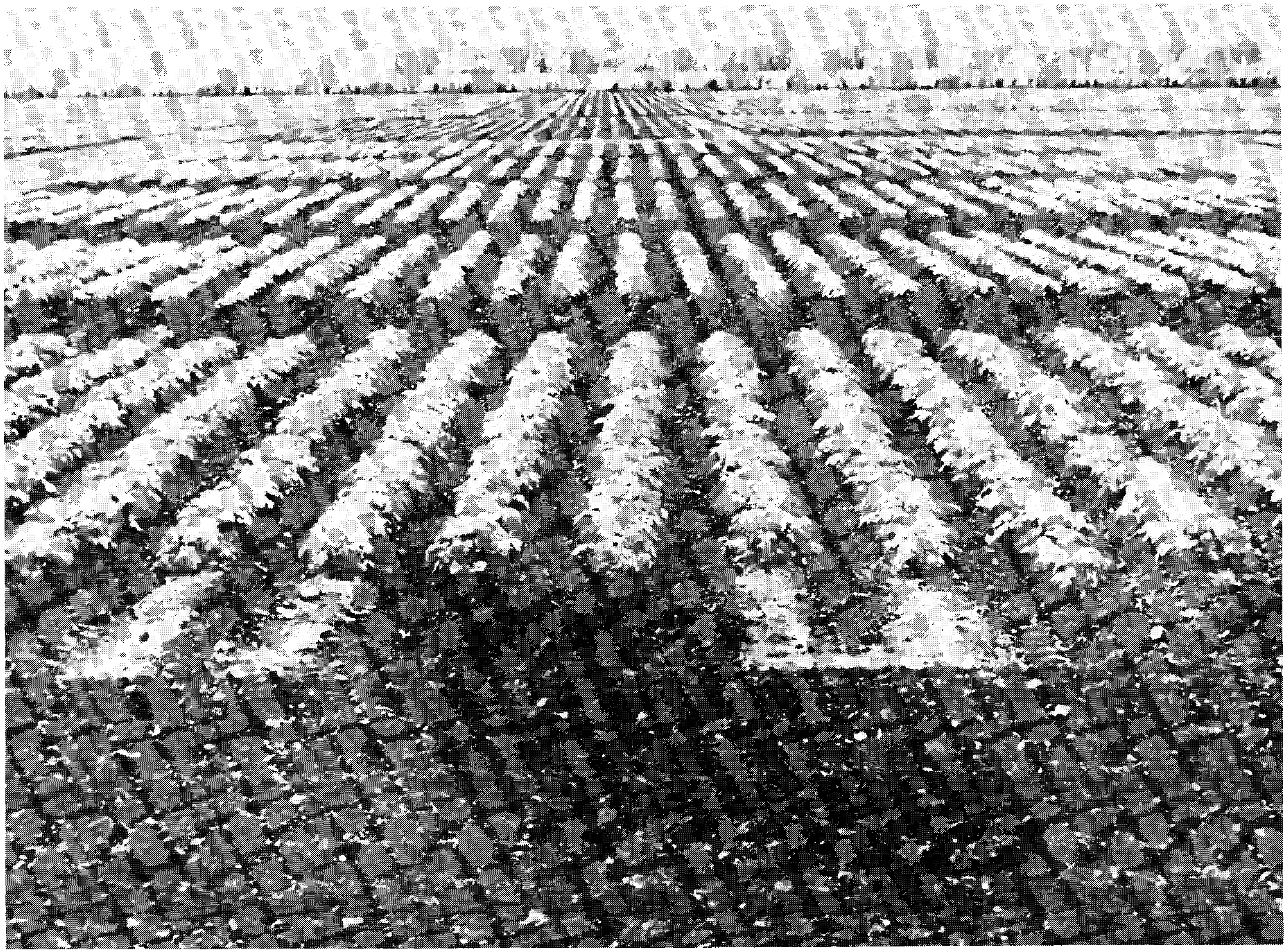

FIGURE 45. - Potatoes in Leonardite-Treated Soil.

minimizing errors due to changes in experimental conditions. The scans are repeated many times to insure statistically reliable results. Typically, about 2,000 scans of the pattern are made over a 3-week period. Printouts of accumulated counts can be made periodically (usually at 12-hour intervals) so that malfunctions will not cause loss of all data.

Analysis of the profiles of peaks in X-ray diffraction intensities is important in structural studies, particularly in revealing crystallite domain sizes and lattice defects. However, such studies have been complicated by effects of experimental factors (usually termed instrumental broadening) on diffracted profiles. A technique has now been developed for correcting such broadening by analyzing the profile of the primary X-ray beam and its wavelength distribution.[63]

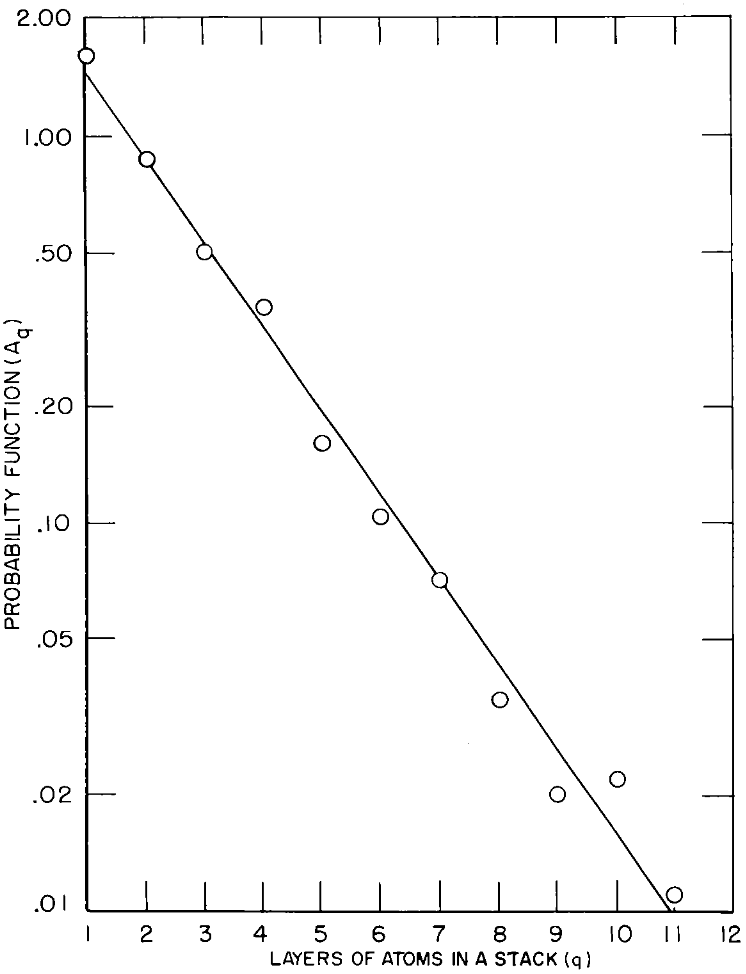

FIGURE 46. - Probability Function for Distribution of Layers in Stacks in Carbon Black.

It is generally believed that most carbons contain structural units or crystallites that consist of more or less planar layers of carbon atoms of differing effective areas, arranged in stacks of differing numbers of layers. Two recent papers have described the results of studies of layer stacking in carbon blacks, as deduced from X-ray diffraction experiments. The distribution of the number of layers in stacks formed by parallel and equidistant layer domains was deduced from the profile of the (002) diffraction peak.[64] The distribution follows the well-known Maxwell-Boltzmann law. Figure 46 shows a plot of a function related to the probability of finding q parallel, equidistant layers in a given stack. The relation has the form $A_q = be^{-aq}$, and the coefficient of the exponent, a, completely defines the profiles of the (002) diffraction peaks. Figure 47 shows observed and calculated

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[63] Ergun, Sabri. Correction of X-Ray Diffraction Profiles for Instrumental Broadening in Transmission Geometry. J. Appl. Phys., v. 40, January 1969, pp. 293-296.

[64] Ergun, Sabri, and Theodore J. Gifford. Stacking Distribution in a Carbon Black. J. Chem. Phys., (n° special), 1969, pp. 99-103.

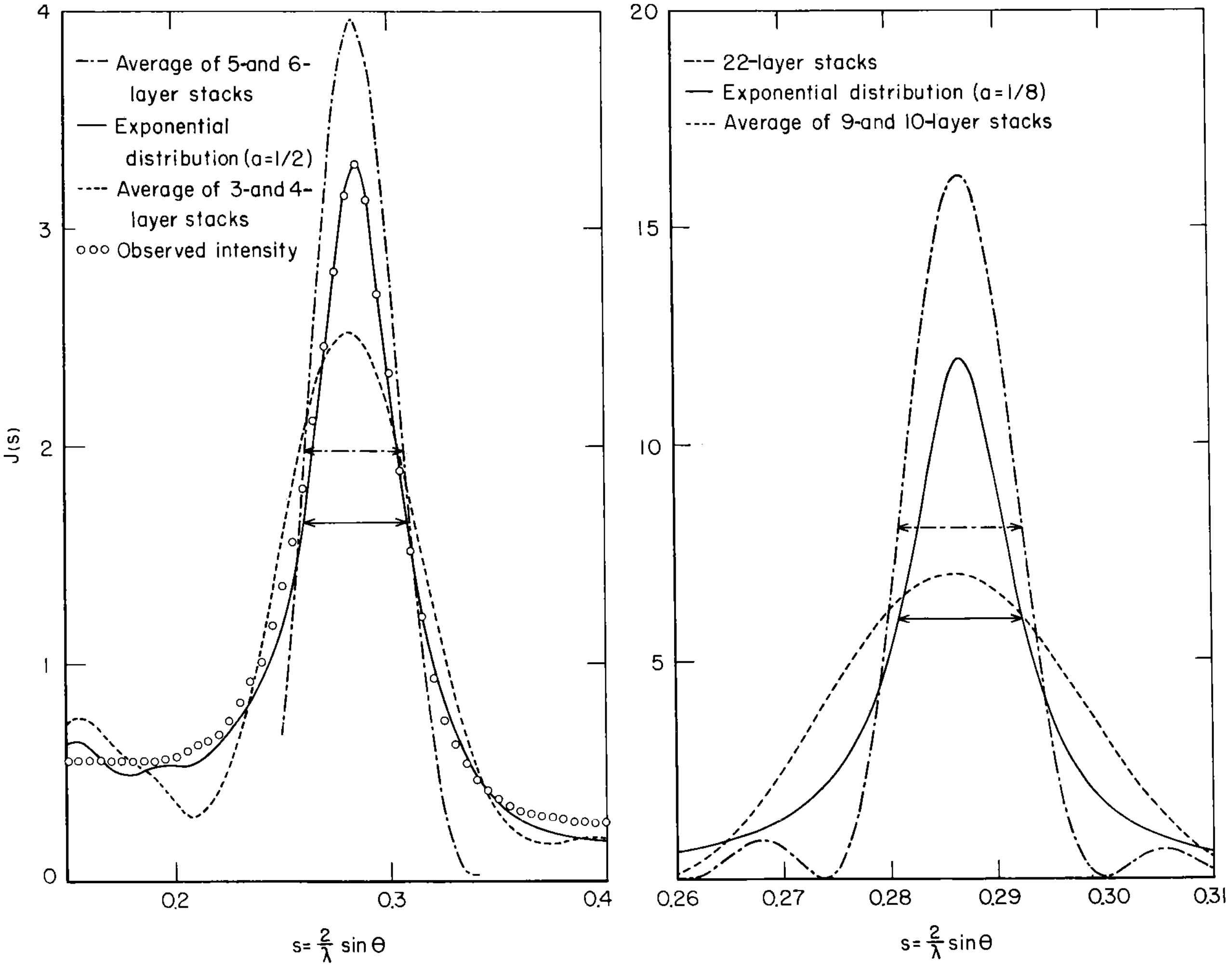

FIGURE 47. - Observed and Calculated Profiles of (002) Reflections of Carbon Black (Left), and Calculated Profiles for Assumed Distributions (Right).

(002) profiles for exponential distributions in stacks of a carbon black, compared with calculated profiles for some assumed assemblages of nearly uniform stacks. A similar study was made of a heat treated black (2,000° C or 3,630° F), it was found that the exponential law is still valid when stacking is extensive.[65]

Proton magnetic resonance (^{1}H NMR) data on coal extracts were extrapolated to estimate the aromaticities of whole coals; agreement with other aromaticity estimates was good. Further, four coals of different rank were examined by broadline carbon-13 magnetic resonance (^{13}C NMR) spectrometry, which required long-term time averaging. Adverse instrumental line broadening has been diminished. The chemical shifts in meta-anthracite appear to be influenced by electrical conductivity as well as by molecular structure.

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[65] Ergun, Sabri, and Theodore J. Gifford. Analysis of the (00ℓ) Reflections of Carbons. J. Appl. Cryst., v. 1, December 1968, pp. 313-317.

Electron spin resonance (ESR) spectrometry was applied to coal extracts, a weathered road tar, high-sulfur coals, high-oxygen coals, and selected humic acids to determine the effect of hetero atoms on ESR g values (g = spectroscopic splitting factor). The free radical electrons in low-rank coals appear to be partially localized on oxygen or sulfur atoms. A small anisotropy of the ESR g value observed in anthracite indicated the presence of graphitelike layers.

Fluorescence spectra of pyridine extracts and carbon disulfide extracts of coals revealed a fluorescence band in the yellow region of the spectrum for each extract. Fluorescence spectra showed greater promise for coal-structure studies than did visible absorption spectra. These are the first such studies on coals. Attempts to observe the laser-Raman spectra of solid and dissolved coal extracts, solid coals, carbons, and graphite were unsuccessful. In the case of the extracts, solid and solution, fluorescence interference proved overwhelming. The excitation fluorescence spectra of an extract indicated that an ultraviolet light source is more suitable for Raman work on coal.

The importance of long reaction times in the coalification process has been demonstrated in the laboratory. Samples of cellulose and pine sawdust heated in vacuo at 200° C (392° F) for a few hours produced very little chemical change. After 2 years at these conditions, however, infrared and ESR spectra of the pine sawdust char and of Dietz subbituminous coal were nearly identical (fig. 48). A temperature of 200° C is considered reasonable for the coalification process, hence the coalification obtained in only 2 years supports the hypothesis that geologic time would produce all ranks of coal at temperatures below 200° C.[66]

An automated reflectance scanning microscope system has been developed, primarily for determining volume proportions, size distribution, and mode of association of the physical (petrographic) components of coals.[67] The process is potentially more rapid and objective and less tedious than a visual microscopic procedure. The technique involves magnetic tape recording of several million reflectance readings on micron-sized areas of polished coal surface, plus computer analysis and interpretation of some characteristics of the coal components. In a study of pyrite distribution in steam coals, the application was essentially successful, but sampling problems and defects in polished pyrite surface characteristics prevented completely satisfactory results. Pyrite content and coal-pyrite particle association could be determined nearly as well as by visual microscopy, but size distribution data were less acceptable.

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[66] Friedel, R. A., J. A. Queiser, and J. L. Retcofsky. Coal-like Substances From Low-Temperature Pyrolysis at Very Long Reaction Times. Preprint, Div. Fuel Chem., Am. Chem. Soc., 157th Nat. Meeting, Minneapolis, Minn., April 1969, v. 12, No. 1, pp. 1-7.

[67] McCartney, J. T., and S. Ergun. A Microscope System Using Automated Reflectance Scanning To Study Coal Components. Application to Analysis of Pyrite Distribution. BuMines Rept. of Inv. 7256, 1969, 15 pp.

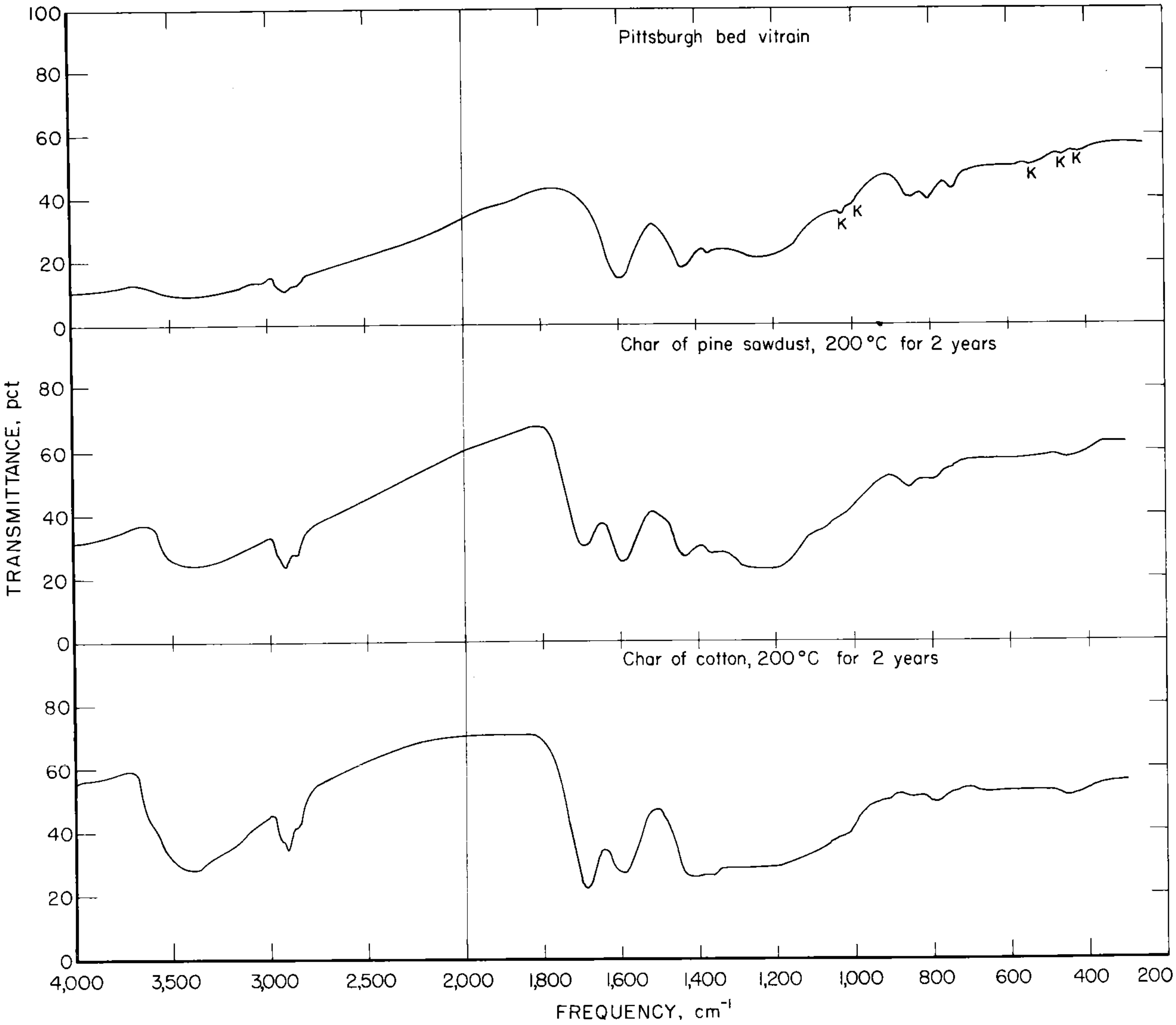

FIGURE 48. - Infrared Spectra of Coal and Simulated Coals.

Organic Contaminants in Wastes

Organic contaminants in airborne particulates and municipal water effluents were investigated by high-resolution mass spectrometry.[68] Treatment of secondary waste water with fly ash reduced the total organic content from

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[68] Sharkey, A. G., Jr., J. L. Shultz, T. Kessler, and R. A. Friedel. Determining Organic Contaminants in Air and Water. Research/Development, v. 20, No. 9, September 1969, pp. 30-32.
Sharkey, A. G., Jr., J. L. Shultz, T. Kessler, and R. A. Friedel. Determination of Organic Contaminants in Air and Water by High-Resolution Mass Spectrometry. Proc. 17th Ann. Conf., Mass Spectrometry and Allied Topics, May 18-23, 1969, pp. 320-322.

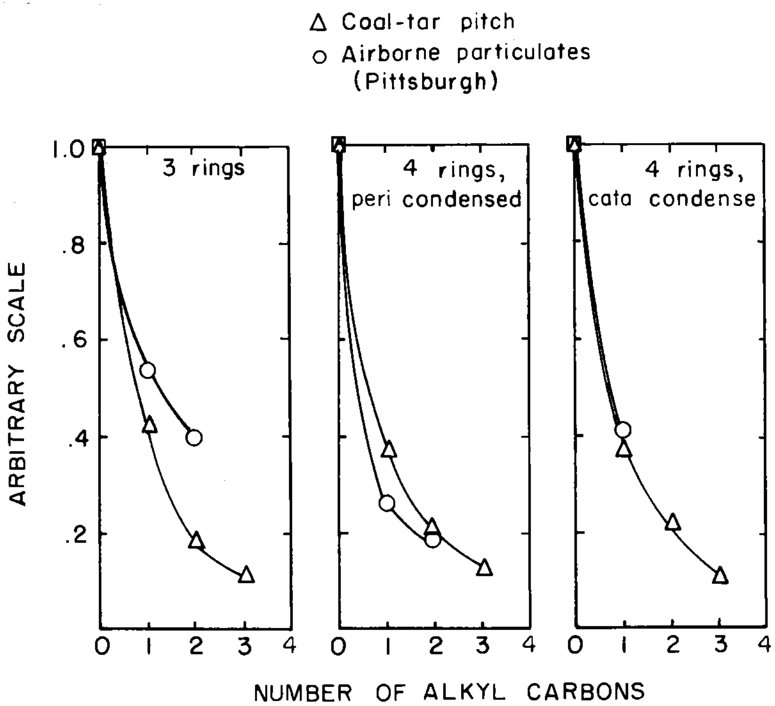

FIGURE 49.-Relative Amounts of Alkyl Derivatives of Polynuclear Aromatic Hydrocarbons in Airborne Dusts and Coal-Tar Pitch.

1.0 part per million to 0.6 part per million. All contaminants detectable by mass spectrometry were removed with approximately equal efficiency.

Organic extracts of airborne dusts were investigated in cooperation with outside laboratories. Molecular formulas were obtained for many oxygenated compounds under suspicion as possible carcinogens. An important finding is that the relative concentrations of alkyl derivatives for compounds derived from airborne dusts and compounds in coal-tar pitch are very similar (fig. 49).

Detection of Inorganics

Electron microprobe work associated with the Mossbauer spectrometry of iron in coal has indicated that iron and silicon occur together in mineral portions of the coal. The mineral, prochlorite, was found to have a Mossbauer spectrum nearly identical to that of the unidentified iron structure in the coal. Usually, however, prochlorite also contains aluminum and magnesium; neither were found.

Coal Ash Constituents

Knowledge of the composition of coal ash is required in most uses of coal. Samples of coal from most commercial beds in 21 States were analyzed for the major constituents: SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, TiO_2, Na_2O, K_2O, P_2O_5, and SO_3.[69] A total of 373 analyses showed that differences in composition can be expected between geographical areas and to a lesser degree in the same bed and

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[69] Abernethy, R. F., M. J. Peterson, and F. H. Gibson. Major Ash Constituents in U.S. Coals. BuMines Rept. of Inv. 7240, 1969, 9 pp.

location. These data may be used for general predictions but not for specific samples.

An examination of 827 samples of coals from commercial shipments in the United States show them to contain 29 elements.[70] These semiquantitative data are correlated with geographical locations and compared with detection limits, distribution, and crustal abundance. It may be used to predict the occurrence of possible air pollutants such as arsenic, beryllium, lead, strontium, and other elements.

SECTION 11. - CHEMISTRY OF COAL

Coal Reactions at High Temperatures

Studies were continued of gaseous and solid products made by laser irradiation of coal and related materials.[71] An important discovery in regard to solid waste disposal is that laser irradiation of coal, oil, rubber, and solid refuse produced a mixture of simple, low molecular-weight gases consisting mostly of hydrogen and acetylene. Continuous irradiation of Pittsburgh seam bituminous coal with a carbon dioxide laser produces a brown solid that is more than 70-percent soluble in benzene, weighs only 0.02 gram per cubic centimeter, and contains more hydrogen than the original coal (fig. 50). Passage of the hot gases from laser irradiation of coal through a bed of Raney nickel catalyst at ambient temperature produced a gas containing more than 90 percent methane, ethane, and propane. In other work, high-resolution mass spectra of gases from the low-energy laser irradiation of humic acids were similar to that from oxidized coal surfaces, giving further insight into the composition of oxidized coal surfaces.[72]

Coal Reactions in Electrical Discharges

Rapid gasification of coal by reaction in electrical discharges to obtain marketable products continued to be studied in 1969. Gasification of coal can be carried out at much lower temperatures by this method than by conventional gasification processes. In addition to hydrogen and carbon monoxide, the usual products, acetylene and methane were also produced.[73] Pyrolysis of coal in a discharge in an argon atmosphere produced similar products;[74] reaction in

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[70] Abernethy, R. F., M. J. Peterson, and F. H. Gibson. Spectrochemical Analyses of Coal Ash for Trace Elements. BuMines Rept. of Inv. 7281, 1969, 30 pp.

[71] Sharkey, A. G., Jr., A. F. Logar, and F. S. Karn. Laser Irradiation of Oxidized Coal. Fuel, v. 48, 1969, pp. 96-97.

[72] Karn, F. S., R. A. Friedel, and A. G. Sharkey, Jr. Coal Pyrolysis Using Laser Irradiation. Fuel, v. 48, 1969, pp. 297-304.

[73] Fu, Y. C., and B. D. Blaustein. Reactions of Coal and Related Materials in Microwave Discharges in Hydrogen, Water Vapour, and Argon. Fuel, v. 47, No. 6, November 1968, pp. 463-474.

[74] Fu, Y. C., and B. D. Blaustein. Pyrolysis of Coals in a Microwave Discharge. Ind. Eng. Chem. Process Des. and Develop., v. 8, No. 2, April 1969. pp. 257-262.

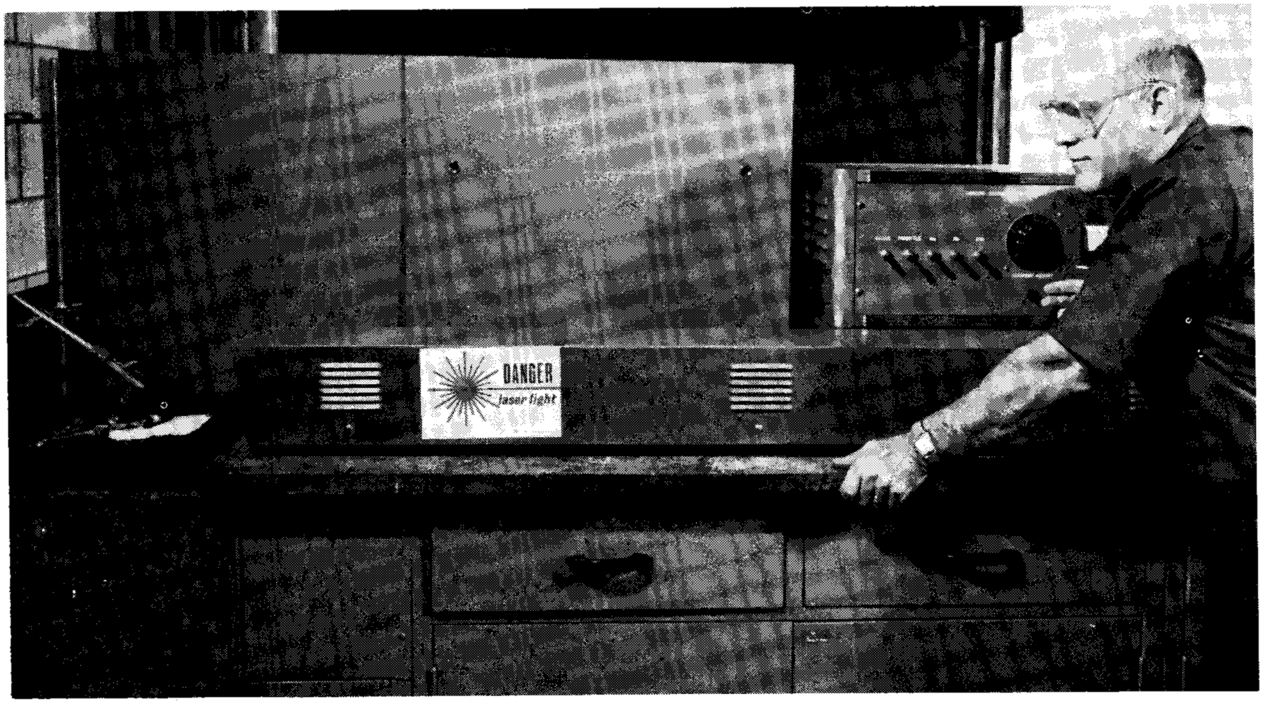

FIGURE 50. - Studying CO_2 Laser Irradiation of Coal.

a nitrogen atmosphere also formed large amounts of hydrogen cyanide. When the gaseous products from the discharge were quickly quenched or removed, such as by trapping at low temperatures, more than 42 percent of the carbon in the coal was converted to hydrogen cyanide and acetylene; these constitute about 90 percent of the total products. Pyrolysis of oil shale, kerogen, gilsonite, and tar sand in a discharge also produced large yields of acetylene and higher hydrocarbons.

Studies are in progress on the hydrogenation and alkylation of coal in a gas-liquid-solid discharge system using tetralin as a hydrogen-donating solvent. Initial experiments with discharged hydrogen and methane as free radical sources indicated that there was some increase in the benzene solubles.

Preliminary experiments were carried on with microwave discharge in a static reactor to produce hydrocarbons for a mixture of hydrogen and carbon monoxide. At pressures of 12 and 50 torr and reaction time of about 1 minute, about 20 percent of the H_2 + CO mixture was converted to methane and acetylene; conversion was increased to more than 90 percent when the reaction products were removed by a cold trap as they formed.[75] These results are being extended to studies in flow systems.

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[75] Blaustein, B. D., and Y. C. Fu. Hydrocarbons From H_2 + CO and H_2 + CO_2 in Microwave Discharges; Le Chatelier's Principle in Discharge Reactions. Ch. in Chemical Reactions in Electrical Discharges. Adv. Chem. Ser. 80, ed. by R. F. Gould. Am. Chem. Soc., Washington, D.C., 1969, pp. 259-271.

Gaseous mixtures of ethylene with air, carbon dioxide, and steam, respectively, were investigated with promising results.[76] A complex mixture of gases and liquids is produced, part of which may prove marketable. Reactions of ethylene and other olefins with steam were continued under investigation. Direct conversion of coal with corona was also attempted, but no conversion could be achieved below about 400° C (752° F). Above 400° C, thermally formed coal tars shorted the electrical corona and produced an arc that interrupted the reaction and damaged the reactor.

Coal Dehydrogenation

Catalytic dehydrogenation of coal in the presence of various vehicles yields almost pure hydrogen gas. Work with bituminlike materials to eliminate the need for more expensive vehicles led to the finding that catalytic dehydrogenation of a petroleum asphalt gave a high-Btu gas consisting of hydrogen and hydrocarbon gases. A high-Btu gas of this type would be valuable commercially. Eight whole coals were catalytically dehydrogenated, and the results were compared with dehydrogenation of vitrinites from bituminous coals. Hydrogen yields were only slightly lower than those from the whole coals.

Work was begun to find cheaper coal-dehydrogenation catalysts than palladium, and cheaper vehicles than phenanthridine. A nickel-on-synthetic-clay catalyst dehydrogenated octahydroanthracene but did not dehydrogenate coal. Evidence was obtained that the sulfur in the coal might have poisoned this catalyst, although it is known that sulfur does not poison other nickel catalysts. A nickel-on-silica-alumina catalyst (potentially a cracking catalyst) was moderately effective for producing hydrogen gas from coal when the nitrogen-containing phenanthridine was used as the vehicle, but not when the higher boiling hydrocarbon pyrene was the vehicle. Fly ash was found to have no catalytic properties for dehydrogenation.

Coal Hydrogenation

A novel method for hydrogenating coal was discovered by the Bureau in 1968, in which synthesis gas (carbon monoxide plus water) is used instead of hydrogen. More recently, some of the operating variables in this process were investigated to provide data for the design and operation of a continuous unit. The mechanism of coal solubilization was also investigated to improve this method of hydrogenation. The effectiveness of synthesis gas was found to be directly proportional to its CO content. Initial gas pressures of 2,000 psig were necessary to obtain 90-percent conversions of lignite with synthesis gas containing 1/2 part CO to 1 part H_2O. The asphaltene content of the product decreased as CO was replaced by H_2, but the decrease in conversion resulting from the lower partial pressure of the CO precluded this method of reducing the asphaltene content. Increasing the temperature from 380° C (716° F) to

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[76] Ruppel, T. C., P. F. Mossbauer, and D. Bienstock. The Effect of a Siemens Ozonizer Discharge on the Reaction of Ethylene and Steam. Div. Fuel Chem., Am. Chem. Soc., 158th Nat. Meeting, New York, Sept. 7-12, 1969, v. 13, No. 4 pp. 313-320.

the 400° to 420° C (752°-788° F) range also decreased the asphaltene content without significantly changing the conversion.

Tar and pitch from the low-temperature carbonization of Texas lignite were satisfactory solvents for the liquefaction of lignite with carbon monoxide (CO) and water. Conversions of lignite near 90 percent were obtained in 2 hours at 380° C (716° F) with initial CO pressures as low as 1,200 psig and equal weights of lignite, solvent, and water. Isoquinoline was found to be a catalyst-solvent for the liquefaction of coal. Subbituminous coal was converted in 93 percent yield to soluble oil, water, and gas. This conversion took place in 20 minutes at 400° C (752° F) at an initial CO pressure of 1,500 psig in the presence of 0.5 part of isoquinoline and 1 part of water per part of coal.

A comparison of the behavior of H_2 and $CO + H_2O$ in the hydrogenation and/or hydrocracking of a variety of substances showed $CO + H_2O$ to be most effective with carbohydrates.[77] Lignin and cellulose in the presence of catalytic quantities of sodium carbonate were converted to oil. Ammonia and isoquinoline were also catalysts for this reaction.

SECTION 12. - ANALYTICAL AND TEST PROCEDURES

Coal utilization technology is constantly changing, as are methods for evaluating coal and coal products. A biennial worldwide survey of the literature[78] discusses new developments and improvements in test methods reported from October 1966 through September 1968.

Beryllium in Coal Ash

Ashes from 33 different coals were found to contain from 0.0005 to 0.021 percent beryllium. The beryllium appeared to be associated with the organic material. The fact that lower ash coals were higher in beryllium was confirmed by comparison with analyses of float-and-sink samples of high-ash coals. Preliminary tests indicate that the beryllium is retained in the ash at 1,000° C (1,832° F).

Plasticity of Coal

The Davis and Gieseler plastometers for determining the plasticity of coal were in the final stages of automation when further work was suspended.

Minerals in Coal

The proximate analysis of the coal macerals by the standard method requires more sample than is usually obtainable. The tests for volatile

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[77] Appell, H. R., I. Wender, and R. D. Miller. Dissimilar Behavior of Carbon Monoxide Plus Water and of Hydrogen in Hydrogenation. Preprints, Div. Fuel Chem., Am. Chem. Soc., v. 13, No. 4, September 1969, pp. 39-44.

[78] Abernethy, R. F., and J. G. Walters. Solid and Gaseous Fuels. Anal. Chem., v. 41, April 1969, pp. 308R-322R.

matter and ash have been scaled down to semimicro scale using 0.1-, 0.2-, and 0.3-gram samples instead of the standard 1.0-gram sample. Present indications are that reliable results with 0.2-gram samples are feasible for both volatile matter and ash.

SECTION 13. - INSPECTION, SAMPLING, AND ANALYSIS

Sampling, inspection, and analytical services were continued for Federal agencies to assist them in procuring coal on specifications and bids. These services include coal analyses, advise on technical matters relating to coal purchases, coal-sampler training, and help in determining contract awards and implementing the contracts. These services have achieved substantial savings to the Government by making it possible to award coal contracts on a guaranteed-quality basis and to adjust prices for shipments of substandard coals. Coal analyses made in the course of this work are published annually[79] and maintained in a file open to the public.

Procurement of coal for the Government, excluding the Atomic Energy Commission and Tennessee Valley Authority, was transferred in 1969 to the Defense Fuel Supply Center. Purchases were estimated at approximately 6 million tons. Because of air pollution regulations, several Federal plants converted to fuels other than coal, hence requests for analyses dropped below the rate for the preceding year. Tipple samples were collected at 119 mines in nine States. A total of 74 samples were collected in three States for studies being conducted for the Public Health Service on the removal of pyritic sulfur.

Under a working agreement with the Atomic Energy Commission, 550 samples of delivered coal were collected and prepared at the Savannah River Operation Plant, Aiken, S.C. Coal-sampling schools were conducted at Wright-Patterson, Ohio, for the Air Force. At the request of Federal agencies, several installations were visited to inspect coal-sampling procedures and equipment and to recommend improvements in their quality-control programs. Analytical, sampling, and inspection service by the coal laboratory at Anchorage, Alaska, continued to facilitate procurement and quality control of coal purchases by the Government from Alaskan mines. Installations of both the Air Force and Army were visited periodically to inspect sampling facilities, train coal-sampling personnel, and make necessary recommendations to improve coal-sampling procedures.

Analytical services also are provided on a continuing basis in connection with coal reserves studies, preparation, and utilization. Included are analyses of oxides of sulfur and nitrogen by several methods of coal combustion for use in atmospheric pollution research. Coal and coal-mine dust samples totaling 32,000 were subjected to 220,000 determinations; 57,000 determinations were made to facilitate research.

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[79] Aresco, S. J., and J. B. Janus. Analyses of Tipple and Delivered Samples of Coal Collected During Fiscal Year 1968. BuMines Rept. of Inv. 7219, 1969, 31 pp.

SECTION 14. - INTERNATIONAL ACTIVITIES

Foreign markets and world energy trends continued to be observed through economic studies, analyses and reviews, and consultation with coal exporters, foreign importers, consumers, and shippers.

Bureau of Mines representations were made at high-level conferences of the European Economic Community's Energy Directorate, the United Nation's European Economic Commission, and at numerous U.S. interagency meetings involving all Federal agencies concerned with the Government's overall export program and the national balance of payments. Availability of coking coals tightened througout the world and the U.S. coal export trade shifted from declining European shipments to soaring exports to Japan. Against this background, the availability of premium coking coals, the productive capacity of mines producing such coals, and rising costs focussed attention on U.S. and world supply problems.

The objectives of sharing in the increased world market by expanding coal exports, with reasonable stability in prices and transportation charges, led to a series of high-level government-industry and interagency conferences dealing with U.S. coal availabilities. During the year, the Bureau organized conferences and study tours of important foreign missions and delegations, among which were a Soviet coal mining exchange delegation headed by a Soviet cabinet member, and a Belgian fact-finding coal mission. Other high-level conferences were conducted with officials of the European Economic Community and with special foreign private industry representatives to discuss trends and developments in coal prices, coal research, rail rates, and supply prospects over the long and short term. Also, the Bureau continued to cooperate with the Agency for International Development and the United Nations by implementing training programs in all phases of coal technology and health and safety during 1969. Eight Pakistani trainees were with the Bureau at various times during the year to study U.S. coal mining practices so that they could better assist private sector industry in Pakistan in developing the country's coal mines. Two mining engineers from Indonesia spent the year at Bureau facilities and with the coal mining industry, and five Polish nationals arrived to study the processes for converting coal to liquid fuels.

Identification of the problems and goals of coal export expansion emphasized the responsibilities of the Department of the Interior vis-a-vis the direction of coal export expansion and related activities.

APPENDIX A.--BUREAU OF MINES ACTIVITIES CONTRIBUTING TO THIS REPORT

Bureau of Mines station	Location	Main coal activities
	HEADQUARTERS ACTIVITIES	
Minerals Research:		
Coal Research	Washington, D.C.	Programing and budgeting, economic analysis, international activities, Government fuel-engineering service, inspection and sampling of coal, boiler-water research and service, inspections and investigations in mines and related plants, safety educational work, mine fire control, collection and analysis of coal mine accident-injury data.
Mining Research		
Mineral Resource Evaluation:		
Division of Mineral Studies		
Division of Environmental Activities.		
Division of International Activities.		
Division of Economic Analysis		
Division of Statistics		
Health and Safety:		
Division of Coal Mine Inspection		
Division of Accident Prevention and Health.		
	FIELD ACTIVITIES	
Minerals Research:		
College Park Metallurgy Research Center.	College Park, Md.	Coal microbiology and metallurgy.
Explosives Research Center	Pittsburgh and Bruceton, Pa.	Explosions, explosives.
Grand Forks Coal Research Laboratory.	Grand Forks, N. Dak.	Lignite preparation and combustion, fouling properties of lignite ash, carbonization of Western coals, nonenergy uses for lignite.
Morgantown Coal Research Center.	Morgantown, W. Va.	Analysis, preparation, carbonization and combustion, electric generating systems, solid wastes utilization, low-temperature coal tar, gas producer, stack-gas purification.
Pittsburgh Coal Research Center.	Pittsburgh and Bruceton, Pa.	Preparation, combustion, stack-gas purification, carbonization, nonenergy uses, liquid and gaseous products by hydrogenation and catalytic synthesis, physical and chemical properties, coal analysis.
Denver Mining Research Center	Denver, Colo.	Mining, ground control.
Mine Systems Engineering Group	do	Mine systems development.
Pittsburgh Mining Research Center.	Pittsburgh and Bruceton, Pa.	Mining, coalbed degasification, acid mine drainage control.
Mineral Resource Evaluation:		
Environmental Affairs Field Office.	Wilkes-Barre, Pa.	Mine drainage and flood control, mine fires, subsidence and surface reclamation, mine data microfilming, and mine-waste disposal.
Pittsburgh Office of Mineral Resources.	Pittsburgh, Pa.	Resources investigations.
Knoxville Office of Mineral Resources.	Knoxville, Tenn.	Do.
Minneapolis Office of Mineral Resources.	Minneapolis, Minn.	Do.
Denver Office of Mineral Resources.	Denver, Colo.	Do.
Alaska Office of Mineral Resources.	Juneau, Alaska	Resources investigations, sampling and analysis.
Health and Safety:		
Health and Safety Research and Testing Center.	Pittsburgh and Bruceton, Pa.	Control of mine fires, roof control, mine ventilation, testing and approval of permissible mining and respiratory-protective equipment, special studies.
Health and Safety District A	Pittsburgh, Pa.	Fire-resistant hydraulic-fluid investigations, mine-rescue communications.
Health and Safety District C	Mount Hope, W. Va.	Gas and mine-dust analysis.
Health and Safety District E	Denver, Colo.	Gas and mine-dust analysis, control of mine fires.

APPENDIX B.--ACKNOWLEDGMENTS

The following organizations, institutions, and companies contributed significantly to the Bureau's coal research and technologic program during 1969. The Bureau gratefully acknowledges this assistance. Some of the groups made contributions under cooperative research agreements; others provided material, equipment, technologic information, or other assistance.

Aluminum Co. of America.
Anthony Mining Co.
Atomic Energy Commission.
Babcock & Wilcox Co.
Basin Electric Power Cooperative.
Baukol-Noonan, Inc.
Bethlehem Mines Corp.
Bituminous Coal Research, Inc.
Blue Coal Corp.
Boich Mining Co.
British Coal Tar Research Association.
British Coal Utilization Research Association.
Bucyrus-Erie Co.
Carnegie-Mellon University.
Caterpillar Tractor Co.
Combustion Engineering Inc.
Consolidation Coal Co.
Continental Oil Corp.
D&M Coal Co.
Dravo Corp.
Duquesne Light Co.
Eastern Associated Coal Corp.
Elkhorn Coal Co.
Elliott Co.
Esso Research & Engineering Co.
Everett Coal Co.
Ferris Coal Co.
Firestone Tire and Rubber Co.
Fisher-Scientific Co.
Great Northern Railway Co.
Glen-Gary Corp.
Governor's Science Committee, Pennsylvania Technical Assistance Program.
Gulf Research and Development Co.
Gullick, Ltd., Wigan, England.
Houdry Process and Chemical Co.
Howe Coal Co.
Imperial Coal Co.
Island Creek Coal Co.
James Bros. Coal Co.
Jones & Laughlin Steel Corp.
Joy Manufacturing Co.
Kaiser Steel Corp.
Kennecott Copper Corp.
Kerr-McGee Corp.
Knife River Coal Mining Co.
Koppers Co.

Madison Wisconsin Refuse Reduction Plant.
Massachusetts Institute of Technology.
Mechanical Technology, Inc.
Mid-Continent Coal and Coke Corp.
Mine Safety Appliance.
Minerals Transport Inc.
Minnkota Electric Power Cooperative, Inc.
Monongahela Power Co.
Montana College of Mineral Science and Technology.
Montana Power Co.
Montana State University.
National Aeronautics and Space Administration.
National Air Pollution Control Administration.
National Ash Association.
New York University.
Northern Pacific Railway Co.
Northern States Power Co.
North American Coal Corp.
North Dakota State University.
Nuclear Magnetic Resonance Specialities, Inc.
Ohio River Colleries Co.
Old Ben Coal Corp.
Oliver Coal Corp.
Otter Tail Power Co.
Peabody Coal Co.
Pennsylvania Department of Health, Division of Occupational Health.
Pennsylvania State University.
Phillips Petroleum Co.
Pittsburgh Activated Carbon Division of Calgon Corp.
Pittston Clinchfield Coal Co.
Polen Coal Co.
Puskarich Mining Inc.
Red River Valley Potato Growers Association.
Scientific-Pittsburgh.
State of Pennsylvania.
Sun Oil Co.
The Fairfield Engineering Co.
The Wolverine Co.
Thompson Brothers Mining Co.
Union Carbon Corp.
United Power Association.
United States Department of Agriculture.
United States Steel Corp.
University of Illinois.
University of North Dakota.
University of Pittsburgh.
Varian Associates.
Virginia Polytechnic Institute.
West Virginia University.
Westinghouse Electric Corp.--New Products Division.
Wilkes Scientific Corp.
Youghiogheny & Ohio Coal Co.

APPENDIX C.--OTHER PUBLICATIONS

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Coke and Coke Chemicals (annual and monthly).

Coke Distribution: Distribution of Oven and Beehive Coke in 1968 (annual).

Coke Producers: Coke Producers in the United States (annual).

International Coal Trade (monthly).

Pennsylvania Anthracite Distribution: Distribution of Pennsylvania Anthracite for the Coal Year April 1, 1968 to March 31, 1969 (annual).

Pennsylvania Anthracite (weekly).

Peat Producers: Peat Producers in the United States in 1968 (annual).

Weekly Coal Report: Production of Bituminous Coal and Lignite (weekly).

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