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William G. Stratton, Governor
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1958

PETROGRAPHIC AND COKING CHARACTERISTICS OF COAL

Laboratory Study of Illinois Coal Seams Nos. 5 and 6

Charles E. Marshall
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BULLETIN 84

ILLINOIS STATE GEOLOGICAL SURVEY
JOHN C. FRYE, *Chief*
URBANA, ILLINOIS

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FOREWORD

Dr. Charles E. Marshall, distinguished British coal geologist and petrographer and now Head of the Department of Geology and Geophysics at the University of Sydney, Australia, spent the period from March to September, 1955, as a Research Affiliate at the Illinois State Geological Survey.

This bulletin presents results of a laboratory investigation, made during that period, which was undertaken primarily to examine the influence of petrographic factors on the character of cokes produced from Illinois coal. A number of specialists on the Survey staff have contributed to this investigation, but techniques used and some terminology in the report differ from current Survey practice.

We plan to explore further some of the apparent trends and some of the factors that influenced the character of the cokes produced in this intensive, though preliminary, investigation. We are now in the process of planning pilot-plant research to determine whether any of these trends might be translated into commercial practice. Close integration of petrographic studies with chemical and physical test data offers promise of yielding valuable information in regard to utilization of Illinois coals.

—JOHN C. FRYE
Chief

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PETROGRAPHIC AND COKING CHARACTERISTICS OF COAL

Laboratory Study of Illinois Coal Seams Nos. 5 and 6

CHARLES E. MARSHALL, JOHN A. HARRISON, JACK A. SIMON,
AND MARGARET A. PARKER

ABSTRACT

A laboratory investigation of petrographic constitution and other factors that influence coking character of southern Illinois Herrin (No. 6) and Harrisburg (No. 5) coals has demonstrated that the petrographic composition of the coal may be an important influence on its coking characteristics. Other factors investigated which influence coking character were: charging temperature, final coking temperature, final coking period, particle size and size distribution, and influence of a benzene soluble coal tar extract ("resin" or "asphaltene") when blended with coal.

Special laboratory coking procedures and tests for shatter, tumbler stability, and hardness were developed for this study. Chemical analyses were made on coals and cokes. Fluidity values and swelling indices were determined on the coal samples. Results of exploratory tests resulted in the selection of a heating rate of 3.6°C per minute, a final coking temperature of 1010°C, and a final coking period of two hours. The standard charging temperatures were 450°C for the No. 6 coal and 540°C for the No. 5 coal, values within and slightly above the plastic ranges of these coals, respectively.

Tests made to determine optimum particle size distribution in the coal charge indicated that it was desirable to use a method of selective crushing which permitted progressive withdrawal of undersized material to prevent overcrushing.

Detailed petrographic analyses made on all coal samples and sized fractions of the coal were in terms of quantity and apparent thickness of vitrinite, exinite, inertinite, and mineral matter. These analyses demonstrated a systematic variation attributable to preferential breakage of the coarser coal band types and constituents. Investigation of the effects of coal types (maceral quantity and apparent thickness) on coke from No. 6 coal showed that optimum cokes were produced from coal with 87 percent vitrinite and a vitrinite median thickness of 15 microns. A gentle but definite trend in increase of coke strength was noted with increased content of inertinite up to concentrations of the order of 9 percent. Addition of minus 150-mesh fusain in proportions from 5 percent to 15 percent produced improvement in coke strength indices under various conditions of coking and testing.

Addition of "asphaltene" was of limited value when the coking character of the coal was relatively good but the quality of coke was much benefited by addition of "asphaltene" and fusain to the coal.

INTRODUCTION

The need for better development and utilization of the national solid fuel resources is increasingly urgent in all industrial countries. Intensified competition from other sources of power, as well as waning reserves of high-quality industrial coals, have produced critical conditions both in coal-mining industries and in industries that depend upon coal as a raw material, of which one of the most important is the coke industry.

The production of metallurgical coke of optimum quality is an essential factor in modern industrial development. Depletion of reserves of those coal seams formerly considered as providing the best coking coals, the more exacting requirements of modern blast furnace practice, together with an increasingly critical balance of economy have emphasized the need for continued scientific and technological interest in the more effective production of better metallurgical coke from coals formerly considered unsuitable. Although much excellent work has been done in various laboratories, pilot plants, and industrial units, it is believed that more detailed and possibly critical information may yet emerge from suitable fundamental studies.

This investigation was concerned with the determination and evaluation of certain fundamental coal bed characteristics which, together with conditions under which the coals were coked, influenced the properties of the cokes produced. A primary objective was the evaluation of petrographic factors as they may influence coking character under different conditions of coking. In particular the study was directed to the low and medium sulfur areas of the Illinois coal seams Nos. 5 and 6, of the Saline and Jefferson counties, respectively.

Neither seam offers the range of petrographic contrast which would be preferred in an initial study of this kind, but their importance to Illinois is such as to demand attention. The results presented must be regarded as no more than a first assessment of the factors concerned; limitations of time, personnel, and equipment prevented further development of many potentially important lines of investigation which were revealed. Many of the conclusions may be applicable to other coals, particularly those of comparable petrographic and chemical character.

In the short time available for the first laboratory studies, it was necessary to develop equipment and satisfactory methods of preparing the coal for coking and evaluation. It was impossible to produce laboratory conditions and tests that would yield results directly comparable with those of either pilot plant or industrial installation, but for the purposes of the initial studies, the techniques evolved were adequate and the results established trends and relative characteristics.

In certain cases parallel serial studies were made upon identical coal charges, coked in the laboratory furnace and in the pilot oven, and tested respectively by laboratory and industrial standard procedures. Although absolute values differed greatly between the laboratory and pilot scale operation results, similar trends were established.

Although specific evaluation of Illinois coals for the production of metallurgical coke was not the primary purpose of this work, the results of the laboratory studies may indicate probable industrial experience in corresponding circumstances.

ACKNOWLEDGMENTS

To the Illinois State Geological Survey, the senior author is particularly indebted for the opportunity and financial assistance, without which it would have been impossible to devote to the development of this project a substantial part of a year's sabbatical leave from the University of Sydney, Australia, through most of 1955.

We are grateful to our colleagues of the Geochemistry Group for all chemical analyses of coal and coke, and Gieseler and free swelling index determinations. We wish to express our particular indebtedness to the late Frank H. Reed, Chief Chemist, and O. W. Rees, Head of the Analytical Division, H. W. Jackman, Head of Chemical Engineering Section, G. R. Yohe, Head of Coal Chemistry Section, and Emile D. Pierron, Associate Chemist in the Analytical Section. G. R. Yohe prepared the "resin" extracted from pitch and used as an additive in some of the studies.

H. W. Jackman and his associates of the Applied Laboratory made pilot plant coking tests upon specially prepared charges. R. J. Helfinstine's unfailing support and ingenuity eased many difficulties and helped solve many technical problems concerned in adapting and devising equipment.

Throughout the period of investigation colleagues of the Coal Section rallied to our support on all necessary occasions.

To the many members of the State Geological Survey, too numerous to name individually, who by their ungrudging assistance and special skills contributed to the solution of the many daily problems, and who have kindly offered numerous suggestions on this study, we offer our appreciation and sincere thanks.

Special acknowledgement and thanks are due G. H. Cady, Senior Geologist and Head of Coal Section, *emeritus*, for his careful reading of the manuscript and many helpful suggestions. Appreciation is due also to other members of the staff who have critically read and offered suggestions on this manuscript.

We are particularly grateful to Mr. Howard Schulz, Superintendent, and Mr. Charles Jankowsky, Engineer of the Orient No. 3 Mine of the Freeman Coal Company, and to Mr. Dewey Marks, Superintendent of Sahara No. 16 mine, and Mr. Clayton Slack, Chief Engineer of Sahara Coal Company, who assisted in the laborious task of collecting large mine pillars of No. 6 and No. 5 coal for the basic samples of this investigation.

PREVIOUS INVESTIGATIONS

Gilbert Thiessen (1937) presented a review of the situation of the coking industry in Illinois, with particular reference to local economic and technological factors, together with an excellent account of the results of a considerable amount of experimental work both on laboratory and industrial scales. It was considered that by coking with a minimum delay after mining, a satisfactory domestic coke could be produced by appropriate methods. The Illinois coals were not highly regarded as sources of good quality metallurgical coke; minimum mineral impurity was regarded as essential to the production of a stronger and better structure. Excessive shrinkage during coking was held responsible for the highly fractured, small and fingery character of the coke; the addition of non-shrinking carbonaceous material as a means of minimizing these volumetric changes was not considered practicable owing to the low coking power of the Illinois coals.

The use of Illinois coals for the production of metallurgical coke was considered specifically by Reed, Jackman, Rees, Yohe, and Henline (1947). It was concluded that both Illinois No. 6 seam coal from Franklin County low-sulfur area, and Illinois No. 5 seam from Saline County medium-sulfur area, could be used continuously in blends with Eastern coals for coking in modern slot-type ovens with the production of an acceptable metallurgical coke. Recommendation not to use Illinois fines for coking was based upon the tendency of fusain to concentrate therein and their greater response to weathering. The results embodied in the report by Reed et al. are both extensive and comprehensive, being based upon laboratory and pilot plant investigations with benefit of cooperation from commercial producers of metallurgical coke.

Cady (1952) has presented a comprehensive summary of the minable coal reserves of Illinois, together with pertinent comments upon coal characteristics.

The blending of Illinois and Eastern coals for the production of metallurgical coke was reviewed in the light of Gieseler plastometer data by Reed, Jackman, Rees, and Henline (1952). Further discussion of plastic and swelling properties was provided by Rees and Pierron in a later report (1954).

Jackman, Eissler, and Reed (1955) reported that insofar as the production of metallurgical coke was concerned, studies on double-screened sizes of southern Illinois No. 5 and No. 6 coals indicated that they were uniform in composition and coking properties. Both coal seams were assessed and compared, briefly but critically, as components in coal blends for the production of metallurgical coke.

The results of more recent studies upon the blending qualities of the Illinois No. 5 and No. 6 seams when used in conjunction with eastern coals have been reported by Jackman, Eissler, and Reed (1956). The effects of adding anthracite fines and coke breeze to blends of Illinois and Eastern coals have been investigated by Jackman, Henline, and Reed (1956) and are of considerable interest in relation to certain results of the present study.

PROCEDURES

Coal Samples: Collection and Selection

The satisfactory evaluation of the coking potentialities of any coal seam requires co-ordination of petrographic, chemical, and coking studies. The studies must be directed not only to proportionately representative seam samples but also to selected coal types or sections of the seam profile. Besides a basis for assessment of the over-all seam coking characteristics, the results of such detailed study provide a basis of evaluation of the extent of variation that may be expected or induced by methods of coal preparation.

For the purposes of such detailed seam, type, and section investigations, random or bulk samples are neither appropriate nor adequate. In order to assess the significance of the results, it is essential that a pillar section of the entire seam be obtained, from which the over-all seam samples and specimens for detailed study may be prepared.

A complete 2-foot square section of the full height of No. 6 coal seam (9 feet thick) was obtained with only minor fracturing along either bedding or joints and without significant disturbance.

The pillar of the No. 6 coal was taken from a fresh face in the Freeman Coal Co. Orient No. 3 Mine, Jefferson County (pl. 1-B). By means of a universal cutter, two vertical cuts were made in the coal face to a depth of rather more than two feet, and separated by approximately two feet of coal; the vertical cuts entered both roof and floor. Previously prepared siding, constructed of one-inch lumber, was used to enclose the three sides of the still attached pillar, and fixed in position by nailing to each other, wedging, and clamping with heavy-duty C-shaped steel brackets. The enclosed pillar, supported by the reinforced wooden siding, was held in position by props, and was then detached by cutting in the roof shale over the coal and then undercutting the floor which caused the back to "shear" and break along the joints. The pillar and supporting trough were then lowered to the floor by appropriate tackle, the enclosing heavy case being completed after packing with damp sacks.

A slightly smaller pillar of the No. 5 coal was obtained about 20 inches square and the full height of the seam (91¾ inches) from the Sahara Coal Co. No. 16 Mine, Saline County (pl. 1-A).

At a distance of approximately two feet from the corner of a freshly dressed crosscut nearest the face of a working room there was drilled in each face a vertical succession of closely spaced holes which intersected at the back of the coal column or sample pillar over the full height of the seam. The two exposed sides of the still attached pillar were supported by previously prepared wooden sides, nailed to each other and secured to the pillar via the drill holes by steel clamps and chain fastenings. The whole was secured by tackle and props. At this stage the pillar was detached by shearing the roof and floor rock, and "picking" out the coal between the adjacent drill holes in each vertical series. It was then lowered to the floor by means of the supporting tackle, and the completed crate was packed with damp sacks.

During the extraction of each pillar, large blocks of the immediately adjacent coal were detached over the full height of the seam. These blocks were packed in large, heavy-duty metal drums closed by tightly fitting lids. These blocks provided a representative bulk sample from which additional sample material could be drawn for either pilot oven tests or laboratory study.

The representative pillars were sent to the laboratory and final samples prepared promptly so as to have minimum moisture loss and oxidation.

In the laboratory, immediately upon uncrating, the macro-petrographic log of the seam was prepared as the basis for the final selection of the type and seam section samples.

The No. 6 coal pillar was divided into four columns by using a wide bladed chisel and hammer. The first column was preserved in correct stratigraphic order and orientation and was used in the preparation of the polished blocks and thin sections required for more detailed petrographic study and control.

The second column was broken and formed the bulk representative reference pillar (RP) sample for the detailed laboratory and coking procedures and provided the principal standard of reference to which other study results were generally referred.

The third column was separated into four benches or seam sections in order to test for the possibility of significant vertical variation of coking character in the seam. The basis for this benching of the pillar was general over-all character or aspect.

The fourth column was divided into coal types based on the thickness and distribution of the vitrain bands as selected by megascopic or low-power microscopic examination.

The coal types were designated *fine clarain* (FC) which was finely laminated and contained vitrain bands up to 1/32 inch thick; *medium clarain* (MC) with vitrain bands in the order of 1/16 inch thick; *coarse clarain* (CC) which contained greater concentration of

wider vitrain bands commonly from ⅛ to ¼ inch thick with occasional bands up to ½ inch; and *"vitrain"* ("V") or *high-vitrain clarain* (HVC) obtained by cutting plus ½-inch vitrain bands from the column with a carborundum saw. As the sheets of vitrain showed imperfect separations along the bedding plane and as it was impossible to cut them out without contamination, the sample was not regarded as pure vitrain.

In the No. 5 coal pillar, petrographic contrast was so poorly defined that the preparation of coal-type samples was not attempted as was done for the No. 6 coal pillar. Three columns were selected from the No. 5 pillar similar to the other three selected for No. 6 coal. The high concentration of sedimentary impurities and pyrite in well defined bands and lenses in the lower 17¾ inches provided a basis for dividing this bed into two seam sections.

All remaining coal of the No. 5 and No. 6 pillars was returned to the respective representative bulk samples that were stored in steel drums for laboratory reserve or for pilot oven coking.

In addition to the samples already described, bulk aggregate samples of fusain were hand-picked from the immediate pillar environment in each seam. In both seams the development of local lenses of fusain aggregates up to two inches thick facilitated collection of this important constituent. In making tests involving addition of fusain, the fusain coal samples used were always from the same bed.

Initial Standard Preparation

Preliminary studies of laboratory equipment and materials indicated that the maximum particle size desirable in the coal charges was of the order of 3 mm. (⅛ inch). Consequently, a standard preparation procedure was adopted for all samples to ensure that they were reduced to the acceptable size range without unnecessary particle fracturing or crushing which would induce unduly high proportions of fines.

The selected coal sample, broken to convenient size by hand, was passed once through a small jaw crusher (jaw separation approximately ¾ inch). The crushed material was passed over a screen with a mesh opening of 3 mm. (⅛ inch); the undersized material was reserved as the first contribution to the final sample.

The oversize was slowly passed through a small set of roll crushers set at a roll separation of 3 mm., and again screened at 3 mm. The undersize was added to the final sample and the oversize repassed through the roll crusher set at 3 mm. This cycle was repeated three times after which there remained only a small proportion of the original sample which had not passed through the 3 mm. screen. This latter fraction, normally of the order of 0.1 to 0.2 percent, consisted of flat particles of such fissile material as argillaceous durain and carbonaceous shale; it was reserved for individual examination. So far as practicable all fly dust produced during this initial preparation of each main sample was carefully collected by hand or vacuum and added to the prepared bulk. The relatively uniform size distribution thus obtained was considered as the standard and is referred to as the "standard size consist."

Each of the crushed bulk samples of coal originally selected from the pillars, after thorough mixing, was divided by quartering into laboratory sample units, each of approximately 1,000 to 1,500 grams weight, and stored until required

in new, air-tight cans of one-half gallon capacity. All samples were used as soon after preparation as was practicable so as to reduce effects due to oxidation and other possible changes.

Size studies of these standard preparations demonstrated an acceptable degree of uniformity within the sample group of the No. 6 coal (table 2). For some reason not clearly evident, the reference pillar of seam No. 5 suffered appreciably greater degradation than either of the component seam sections as indicated by the high proportion of fine size material (table 19). This may have been due to the greater friability of high-vitrain concentrations in the presence of the tough clay and pyritic partings which constituted much of the lower bench of the seam. The relatively lower vitrain content of the bottom seam section (SS I) together with the higher content of more resistant, tough mineral and rock partings, would tend to mask any similar trend in that bench when crushed alone.

Petrographic Examination, Analysis and Assessment

Coal Column

In addition to the initial macroscopic log prepared from the freshly broken surfaces of the reference pillar, polished blocks covering the full thickness of each of the coal seams were examined with oblique illumination using a stereobinocular microscope with magnifications up to 30×. Thin sections were prepared from selected parts of the column as an additional check upon the type identification, character, and distribution as established by both macroscopic appraisal and microscopic examination in the polished blocks.

Broken Coal Samples

For the purposes of petrographic control and critical analysis of the results of the coking tests, detailed micrometric studies were completed of every size fraction of each coal type or seam section sample used in the investigation. Material selected as representative of the broken coal size fractions of each sample was mounted in paraplex (Paraplex "P" series resins). A flat section was ground and polished on the broken coal mount, providing a series of microsections of coal fragments in the size fraction, all of random orientation.

Coal was mixed with equal amount of paraplex by volume in a round glass mold having an inside diameter of 1⅜ inches. After thorough mixing, the "block" was cured in an oven at 90°C. for 8 hours.

After curing, a flat surface was ground using a No. 3 dry emery paper. A finer 3/0 dry emery paper used in the second grinding produced a flat, partially polished surface. The third step was carried out on a polishing wheel covered with "metcloth" (nap-free cloth) and using an aqueous suspension of No. 1 alumina. This was followed by polishing the block on another metcloth on which a finer No. 3 alumina was used with a minimum amount of water. In the final stage the specimen or block was polished on a high quality billiard cloth over which a fine stream of distilled water was running. This method produced a highly polished scratch-free surface.

The method of petrographic analysis was designed to provide information not only upon the volumetric proportions of the seam constituents (macerals and minerals) but also upon their mean width, as both these factors affect the petrographic makeup of the coal and coke oven charge.

For this investigation, the macerals of the coal were designated and measured under the group terms of vitrinite, exinite, and inertinite. Vitrinite included all vitrain bands with the lower size limit imposed by the resolving

power of the microscope, humic degradation matter, resin rodlets, and resins classified as red resins in the transmitted light studies. All spore coats, cuticles, and yellow resins were considered as exinite. Under the term inertinite were grouped the macerals known as fusinite, semi-fusinite, micrinite, and a maceral that resembled sclerotinite. Mineral matter was also determined.

Because of time limitations, the microscopic (or micrometric) analyses were restricted to the broken coal, thus providing analyses of the actual material used in coking tests. Maceral distribution in the individual coal types and proportions and width of the coal microtypes as they occurred in the original pillar were not determined in the present investigation. The value and reliability of the results afforded by these particular methods have been demonstrated in practice over many years (Raistrick and Marshall, 1939).

All analyses were made using a binocular metallurgical microscope equipped with an eyepiece micrometer scale in one of the 8× oculars, and an 8 mm. oil immersion objective. Making allowance for the correction factor of 1.6× for the binocular body, the analytical measurements were made at a magnification of 320 diameters.

Three traverses, each 2 cm. long and spaced 8 mm. apart, were made on every fraction mount. Each fragment of coal encountered was oriented so that the direction of traverse was normal to the banding and the intercept thickness of each constituent (maceral) or mineral was recorded individually in microns in sequence. Although it was recognized that thickness measurements for each group maceral by this technique would be equal to or greater than true thickness (never less than true thickness) no correction factor was applied. Results are considered statistically comparable between samples when this technique is used on broken coal. At the conclusion of the study of each coal fragment, the stage was returned to the initial point of intersection with that fragment and the original direction of the traverse was resumed. The process was repeated upon the next fragment and succeeding fragments encountered until a total traverse of 6 cm. was completed.

The micrometric assessment of the mineral matter is naturally less reliable than the determinations of the coal constituents, because much of the inorganic matter may be present as extremely fine, almost submicroscopic mineral, distributed between and within the organic coal constituents. From the final detailed log there was extracted among other factors the volumetric proportions of each coal maceral and mineral matter, their mean size, and size frequency distribution.

From the latter data, cumulative size distribution curves were prepared from which were obtained 25, 50 and 75 percent (median and quartile) size factors. It was considered that these provided better indications of constituent size distribution than the average. Both the maceral volumetric proportion and size distribution data of the respective size fractions of each sample proved significant in interpreting variations in the characteristics of cokes produced from the various samples.

Finally, from the detailed micrometric data of each of the size fractions, the over-all volumetric proportions and group maceral size characteristics were calculated for each of the samples of the two seams as used in the coking tests.

Chemical Analyses, Gieseler Values, and Free Swelling Index

From the great number of pillar, seam, type, and size fraction samples, those considered of particular importance were selected for chemical analysis and determination of plastic and free swelling properties using accepted methods (Gieseler fluidity and FSI). In certain cases, the physical tests following accepted procedures (except for particle size) were performed upon materials as present in the charge to be coked. Differences emerged in results obtained in the two procedures (accepted method and modified procedure) which, although not great, proved of some interest.

Possible influence of oxidation on test results was recognized, but such effects were held to a minimum insofar as possible. No tests were made to determine the influence of oxidation.

Coke Production

In view of the short time available for the study and the primary objective of study of petrographic factors as they influence behavior of Illinois coal in coking processes, it was necessary to adapt available equipment to the purposes of the investigation with such improvised modification as necessary.

It was particularly fortunate that there was available a Harper Globar type furnace with excellent thermostatic control, capable of being raised to much higher temperatures than those required (pl. 1-C). With the addition of a flue and exhaust system for the removal of volatiles, this unit gave excellent service; the rate of volatile removal could be closely adjusted by a system of dampers which prevented appreciable air flow into the furnace and so greatly minimized the risks of oxidation effects during coking.

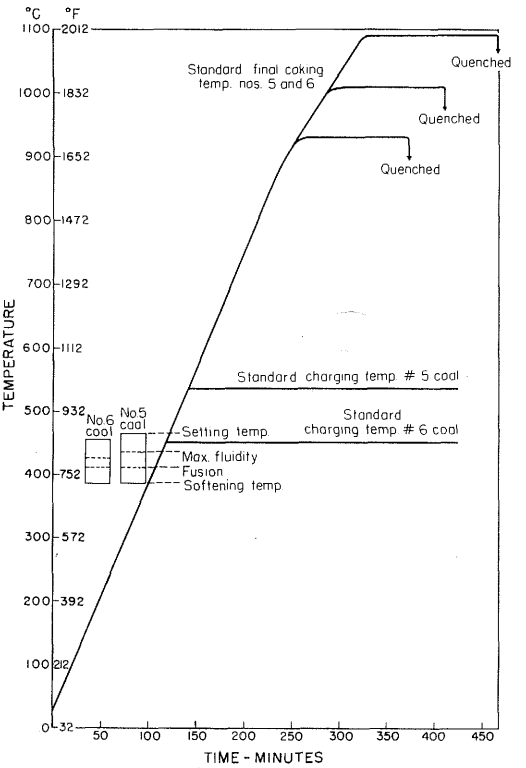
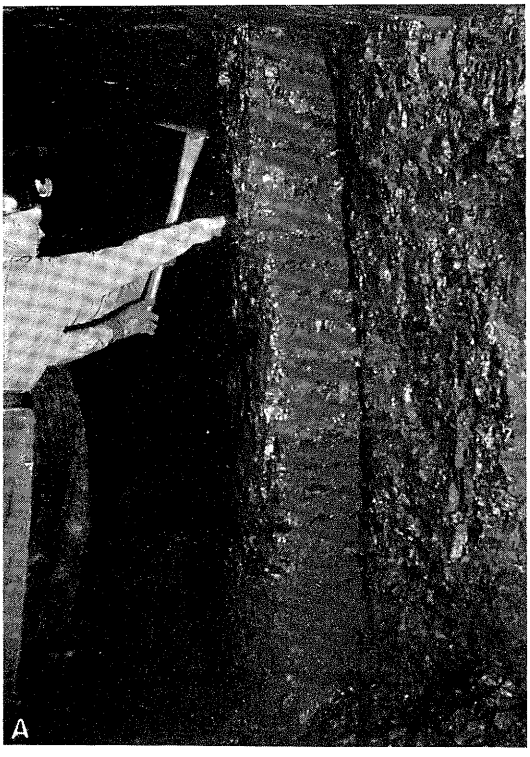
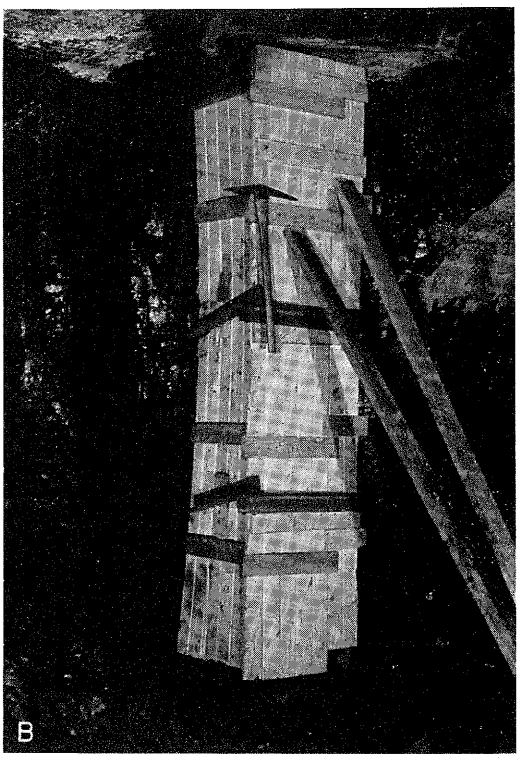
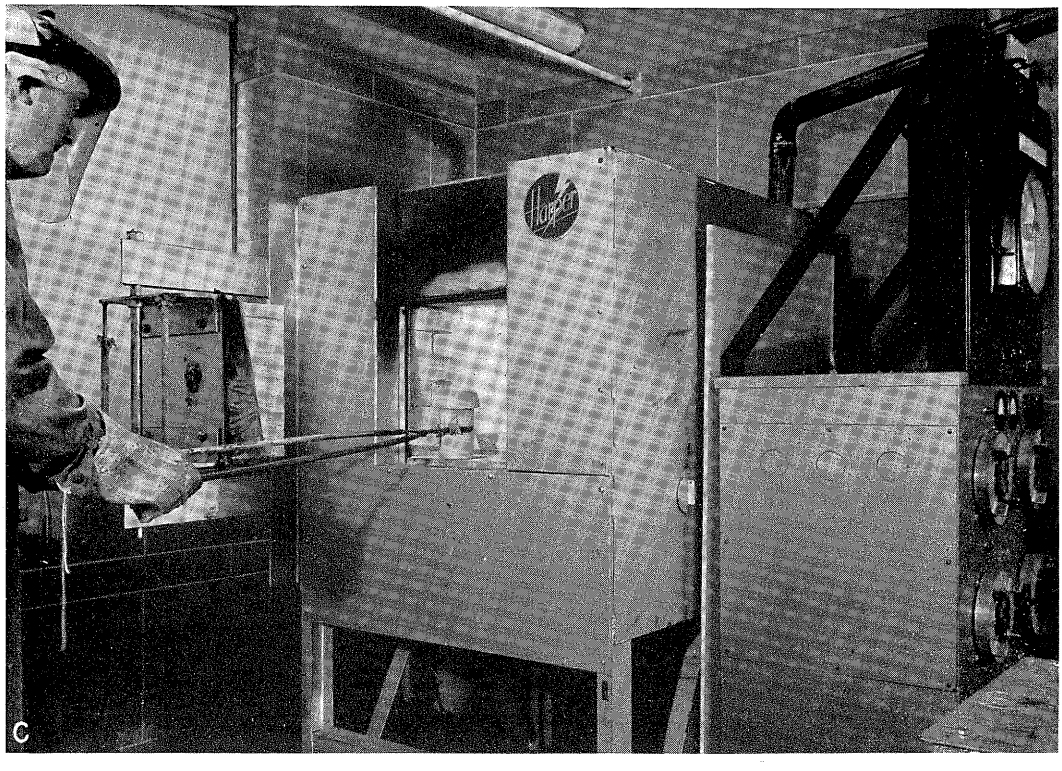


Fig. 1.—Standard furnace heating rate and coking cycles for coals No. 5 and No. 6.

The maximum practicable controlled heating rate for this furnace with its normal content of nine coking retorts proved to be a consistent 3.6°C per minute over the range from room temperature to 900°C; at higher temperatures the rate decreased slightly (fig. 1). Coal in the pilot size coke oven operated by the Illinois Geological Survey increases in temperature as much as 7°C per minute in the early stages of carbonization adjacent to the oven walls, and at about half that rate in the center of the coal charge during the period of maximum temperature increase. The overall heating rate for the complete coking cycle is much lower.







COKING CHARACTERISTICS OF COAL

In consideration of these facts the maximum practicable controlled heating rate of 3.6°C per minute for the Harper Globar furnace was accepted as standard and automatically controlled by special cam. Although this heating rate could not be exceeded except very temporarily, as a result of inadvertent volatile combustion within the furnace (but not the retorts), it would have been interesting, had time permitted, to study the effects of slower rates of heating, particularly through the plastic range of each coal. It is considered that the results of such studies would prove significant.

Preliminary testing was devoted to selecting the most suitable size and type of retorts. As it was intended to examine the cokes by laboratory tests similar to or modeled after full-scale industrial methods, it was essential that the quantity of coke produced should be of adequate proportions, commensurate with the size of the retorts available, the capacity of the oven, and the sample resources. Various sizes of metal, porcelain, fireclay, and alumina retorts were tested in the actual process of coke making. The most satisfactory proved to be of alumina, cylindrical in shape, three inches in diameter, and approximately six inches in height, in which coal charges of 300 to 500 grams could be coked conveniently. Fireclay covers and bases were used to protect the charge and the retort (pl. 1-C).

Each coke run was made in triplicate and, as the oven would accommodate nine retorts, three different coke runs could be undertaken in a single heat. The three retort charges constituting a single run, each of not less than 300 grams weight, were taken from one sealed sample can and given such preparation as the particular test required. The weight of the coal charge was recorded in every case and, depending upon circumstances, the dimensions also.

The coking procedures formed part of the study program and are elaborated in the appropriate sections of this report. They were directed towards determination of the optimum conditions for coking each coal within the limitations of the equipment available. Upon the basis of these results "standard" laboratory conditions were formulated for coking each coal.

At the conclusion of the actual coking process, the retort contents were quenched individually in separate water baths for the minimum time necessary to permit handling and with as little disruption of structure as possible. Thereafter the coke was removed, the fines recovered by decantation, and the whole dried in air for two days on the warm top of the Harper furnace.

Throughout the entire process, systematic record and identification of each unit was carefully maintained.

Coke Tests

The methods of testing evolved for this investigation are modifications of industrial shatter and tumbler procedures, supplemented by an adaptation of the micro-mechanical method developed by Blayden, Noble and Riley

EXPLANATION OF PLATE 1

A.—Pillar of No. 5 coal drilled out of corner of crosscut.

B.—Pillar of No. 6 coal cut from working face, boxed for removal from the mine.

C.—Charging alumina crucible with fireclay cover and base into furnace used for laboratory coking tests.

(1937). All procedures were standardized.

By adapting and modifying the commercial methods to meet the requirements of the laboratory study, it was hoped that the correlation of trends in coke characteristics, if not of absolute values, would be practicable. In those few instances where the results of laboratory tests upon a series of laboratory-produced cokes could be compared with the results of commercial-scale testing of a series of pilot-oven cokes, both series were made from identical charges. The absolute values proved to be quite different but the trends were closely similar.

The combination of tests was considered desirable because the shatter and tumbler test results are largely conditioned by the macro-structure of the coke (joints, cracks, pore dimensions, and wall thickness) whereas the micro-mechanical tests, and to a certain extent the abrasion-fines produced during the tumbler test, are largely conditioned by the strength and toughness of the coke substance.

Before being subjected to shatter, tumbler, or micro-mechanical tests, the coke was sized by a standardized procedure, taking great care to reduce initial breakage. The records were based upon the proportions of plus 1½ inch, 1½ x 1 inch, 1 x ½ inch, ½ x ¼ inch and minus ¼ inch mesh. After sizing, the coke size fractions were recombined and the entire sample was used in the appropriate test.

Shatter Index

In the shatter test the entire coke sample was dropped three times from a height of six feet on to a thick iron plate, through a wide galvanized tube (diameter approximately 12 inches) that was used to avoid scattering of the specimen. The coke was then again carefully sized, and the same size fractions recorded. After recombining the coke and after three additional drops, the coke was finally sized and stored for later reference. The three-stage size study permitted an appraisal of the progressive degradation. The shatter index was recorded as the percentage of plus 1-inch coke remaining after six drops.

Tumbler Stability Index and Resistance to Abrasion

In the tumbler test an entire coke sample was treated for two periods of 20 minutes each in a cylindrical one-half gallon can, with a close-fitting lid, which was rotated at 40 revolutions per minute about a diametric axis. The initial, intermediate (after 20 minutes), and final (after 40 minutes) size consist proportions provided an appraisal of the tumbler degradation characteristics; the stability index was measured by the plus 1-inch percentage and the resistance to abrasion (hardness) by the plus ¼-inch.

Micro-mechanical Strength

The equipment described by Blayden, Noble, and Riley consisted of two stainless steel tubes of 1-inch internal diameter, burnished on the inside and fitted with screw-on steel caps designed to form a dust-proof joint; the effective internal length of the tubes was 12 inches. Twelve steel balls, each 5/16 inch in diameter, were used in each tube to disintegrate the coke.

In practice, 2 grams of dry coke, graded between 14- and 28-mesh Tyler sieves, were used in the tubes which were rotated end-over-end at a constant speed of 25 revolutions per minute for 32 minutes (800 revolutions). The dis-

integrated coke substance was sieved and the coke micro-strength reported in terms of the percentage remaining plus 65-mesh and the ratio of the proportions of the +28/+65 mesh.

These micro-mechanical strength indices are later referred to as "micro-strength 65" and "micro-strength 28/65."

Chemical Characteristics

In a limited number of series where significant chemical variation appeared possible, proximate analyses, total sulfur, and occasionally calorific (Btu) value of the cokes were determined.

Petrographic Examination

The investigation of microstructural characteristics of the coke form and substance is a potentially important study. Examination has so far been confined to special cokes produced from clarain-fusain blends.

STUDY RESULTS

NO. 6 COAL, JEFFERSON COUNTY SEAM AND SAMPLES

The macro-petrographic structure and constitution of the No. 6 coal pillar section used in this study are summarized in table 1. In addition to the characteristic "blue band" (a clay-shale parting 1½ inches thick with silty lenses) the column included a number of shale, clay, and bone (or splint) partings, occasionally persistent but commonly flatly lenticular and erratic in distribution. Pyritic bands developed sporadically along the bedding were not conspicuous except in the upper portion of the seam, where pyrite was also present in joints and vertical fractures.

With the exception of a few minor bands of duro-clarain and durain, the seam consisted chiefly of a medium-grained clarain in which individual vitrain sheets about 1/16 inch thick formed a substantial part of the coal. Locally, greater concentrations of vitrain sheets about ⅛ to ¼ inch thick gave rise to what is considered as coarse clarain; similarly very finely laminated clarain in which the individual vitrain sheets were about 1/32 inch or less thick was accepted as fine clarain. Individual and relatively thick vitrain sheets (up to ½ inch or more thick) occurred sporadically throughout the seam. Fusain horizons were common, occurring as persistent continuous layers of some thickness (⅛ inch) and minor horizons characterized by sporadic distribution of fusain lenticles in variable concentration. In addition to pyrite, carbonate mineral (including calcite) occurred in a large proportion of the joint planes.

Microscopic examination revealed that many of the massive clarains of the seam were composed of thin, alternating, very finely banded clarain and duro-clarain layers, in which occurred occasional thin durain bands with relatively abundant sedimentary mineral matter. Micro-pyrite was evident in clarains and individual vitrain bands.

For the coking studies, in addition to the bulk sample of the reference pillar (RP) there were selected aggregate samples of fine, medium, and coarse clarain (FC, MC, and CC), and high-vitrain clarain or "Vitrain" (HVC or "V").

In order to assess possible significant variation of petrographic character or coking property as related to the seam profile, four section samples were prepared, their boundaries being related to prominent features or partings in the seam as are indicated in table 1.

From all of these samples there were excluded all shale, bone, or pyritic bodies which reasonably could be extracted by hand.

TABLE 1.—No. 6 COAL: MACRO-PETROGRAPHIC ANALYSIS OF COLUMN.

Roof: Gray shale with small and few carbonaceous fragments.

Seam Section IV (SS IV)

3″	Medium clarain with interlaminated clay "films" and granular and submassive pyrite. Very strong parting along bedding.
12½″	Medium clarain with a few vitrain sheets up to 3/8″ thick. Pyrite in thin horizontal partings (<1/16″) and lenticles (10″ x ⅛″) in upper 3″ of the band; also as irregular masses (±6″) occupying local vertical fracture zones.
2¾″	Coarse clarain with many vitrain sheets up to ⅛″ in thickness. Pyrite films in cleat.
⅜″	Fusain: irregular lenticular bodies of contrasting physical appearance.
6½″	Coarse clarain with vitrain sheets up to ¼″ thick. Pyrite in cleat.
1/16″	Fusain: impersistent horizon marked by small lenticular bodies.

Seam Section III (SS III)

10″	Coarse and medium clarain interlaminated with vitrain sheets up to ¼″ thick. Pyrite in cleat and bedding as well as irregular nodules of varied size.
1/16″	Fusain: horizon marked by relatively minor development of small lenticles.
¾″	Coarse clarain with vitrain sheets up to ¼″ thick.
2½″	Medium clarain with vitrain sheets up to ⅛″ thick.
½″	Bone or splint band or carbonaceous shale parting.
6¾″	Coarse clarain with vitrain layers up to ½″ thick.
¾″	Fusain: compact lenticles.
1¾″	Medium clarain with vitrain sheets up to 1/16″ thick.
⅜″	Bone or splint band or carbonaceous shale parting.
2¾″	Coarse clarain with vitrain sheets up to ½″ thick and some lenticular fusain.
1/16″	Fusain: horizon with lenticles up to 3″ x ¼″.
2¾″	Medium clarain with a few vitrain layers up to ¼″ thick.
⅛″	Fusain horizon of relatively fine lenticles.
4″	Medium clarain with a few vitrain layers up to ⅜″ thick.
¼″	Fusain aggregate of finely lenticular material.

Seam Section II (SS II)

1¾″	Medium clarain with vitrain sheets up to 1/16″ thick.
9¾″	Coarse clarain with vitrain sheets up to ½″ thick. Lenticular fusain bodies up to 2″ x ¼″ in lower part of section.
⅜″	Bone or splint band or highly carbonaceous shale parting.
¾″	Coarse clarain with vitrain sheets up to ⅛″ thick.
3″	Medium clarain with well defined bedding.
1¾″	Coarse clarain with fine clarain laminations.
¼″	Clayband; highly carbonaceous.
3¾″	Medium clarain with a few vitrain sheets up to ⅜″ thick.
1″	Clay and massive pyrite horizon of markedly variable and lenticular character.
1¼″	Medium clarain with well defined bedding.
½″	Clay band; irregular in thickness and variable in character.
1″	Medium clarain with well defined, fine vitrain laminae.
1/16″	Fusain: irregular horizon with lenticular bodies of contrasted size and form.
3½″	Medium clarain with well defined fine vitrain sheets.

Excluded

1½″	Blue band; irregular in thickness and containing numerous vitrain laminae often of highly irregular form.

Seam Section I (SS I)

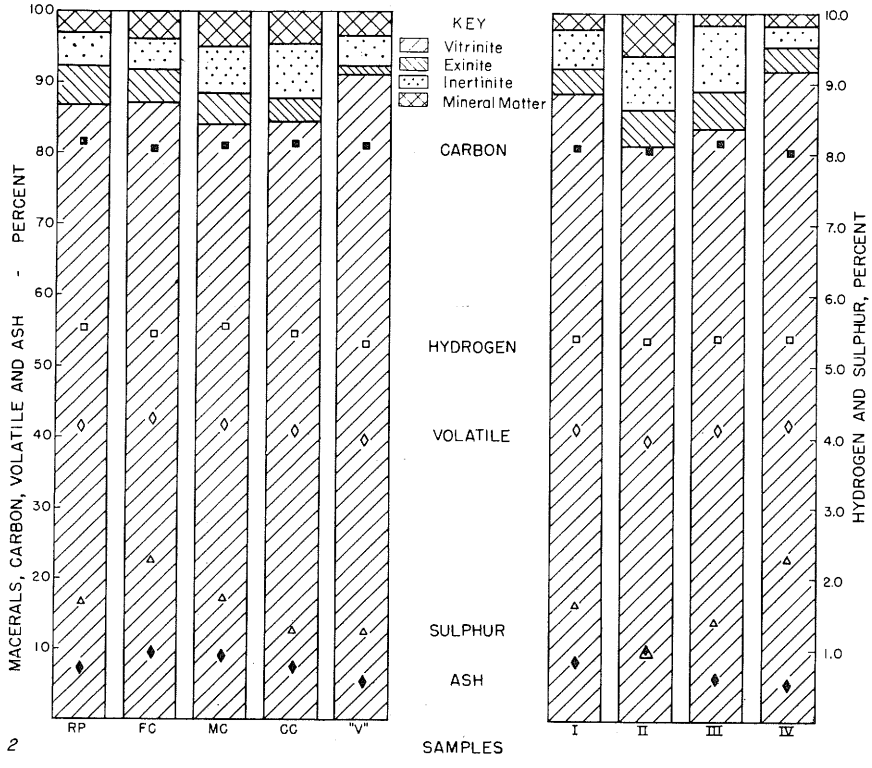
1½″	Coarse clarain with vitrain up to ¼″ in thickness; also lenticular bodies of mineralized fusain (5″ x ½″).
1¾″	Medium clarain of fine to medium vitrain layers.
1/16″	Pyrite horizon; persistent thin sheet of subgranular character.
¾″	Medium clarain with well defined lamination.
½″	Fusain band constituted of lenticular bodies of soft fusain; little evident mineralization.
¾″	Fine clarain and duroclarain closely interlaminated.
11″	Medium clarain with a few vitrain sheets up to ½″ in thickness.
4½″	Fine clarain and duroclarain with a few vitrain sheets up to ½″ thick.
107½″	Total thickness of measured column

PETROGRAPHIC AND CHEMICAL CHARACTERISTICS

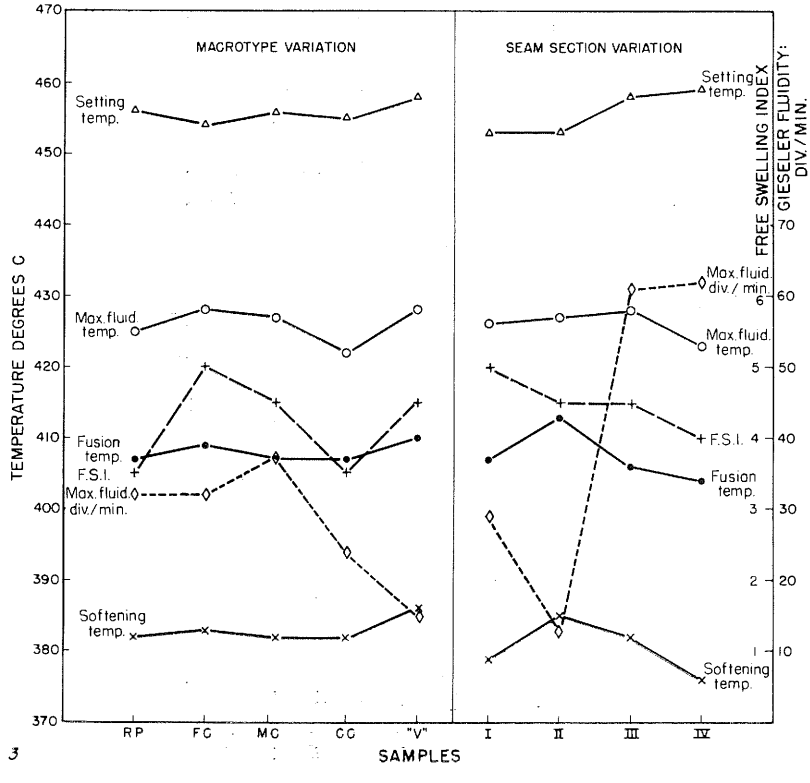
The over-all petrographic and chemical characteristics of the various samples are recorded in tables 3A, 3B, and 4, and are shown graphically in figures 2 and 3. Beginning with the reference pillar as "standard" the samples have been arranged in a series in accordance with macroscopic increase in thickness of vitrain bands, through the fine, medium, coarse, and high-vitrain clarains;

FIG. 2.—No. 6 coal: Petrographic and chemical variations in principal macro-type and seam section samples (tables 3A and 4).

FIG. 3.—No. 6 coal: Variation in Gieseler values and free swelling indices for principal macro-type and seam section samples, minus 6-mesh (table 4).


KEY
Vitrinite
Exinite
Inertinite
Mineral Matter
CARBON
HYDROGEN
VOLATILE
SULPHUR
ASH
MACERALS, CARBON, VOLATILE AND ASH - PERCENT
HYDROGEN AND SULPHUR, PERCENT
RP
FC
MC
CC
"V"
I
II
III
IV
SAMPLES

2


MACROTYPE VARIATION
SEAM SECTION VARIATION
Setting temp.
Max. fluid temp.
Fusion temp.
F.S.I.
Max. fluid div./min.
Softening temp.
TEMPERATURE DEGREES C
FREE SWELLING INDEX
GIESELER FLUIDITY: DIV./MIN.
RP
FC
MC
CC
"V"
I
II
III
IV
SAMPLES

3

TABLE 2.—No. 6 COAL: SIZE ANALYSIS, INDIVIDUAL AND CUMULATIVE, OF REFERENCE PILLAR, COAL MACRO-TYPES, AND SEAM SECTIONS
(Prepared by standard procedure)
In percent

Sieve size (mesh)	Reference pillar		Fine clarain		Med. clarain		Coarse clarain		"Vitrain"		Seam section I		Seam section II		Seam section III		Seam section IV	
	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+6	0.31	0.31	0.17	0.17	0.15	0.15	0.10	0.10	0.17	0.17	0.12	0.12	0.10	0.10	0.15	0.15	0.14	0.14
6 x 10	49.32	49.63	51.00	51.17	47.72	47.87	47.48	47.58	49.69	49.86	47.88	48.00	45.84	45.94	48.89	49.04	45.95	46.09
10 x 20	25.32	74.95	24.67	75.84	25.10	72.97	25.25	72.83	26.73	76.59	25.73	73.73	25.16	71.10	24.91	73.95	26.39	72.48
20 x 35	12.34	87.29	10.99	86.83	12.31	85.28	12.13	84.96	11.99	88.58	11.83	85.56	12.71	83.81	11.50	85.45	12.98	85.46
35 x 65	6.33	93.62	5.76	92.59	6.60	91.88	6.45	91.41	5.53	94.11	6.20	91.76	7.04	90.85	6.06	91.51	6.86	92.32
65 x 150	3.68	97.30	3.42	96.01	3.98	95.86	3.86	95.27	2.82	96.93	3.83	95.59	4.34	95.19	3.60	95.11	3.96	96.28
−150	2.70	100.00	3.99	100.00	4.14	100.00	4.73	100.00	3.07	100.00	4.41	100.00	4.81	100.00	4.88	100.00	3.72	100.00

TABLE 3A.—No. 6 COAL: MICROMETRIC (PETROGRAPHIC) ANALYSES OF MINUS 6-MESH FRACTIONS
(Prepared by standard procedure)

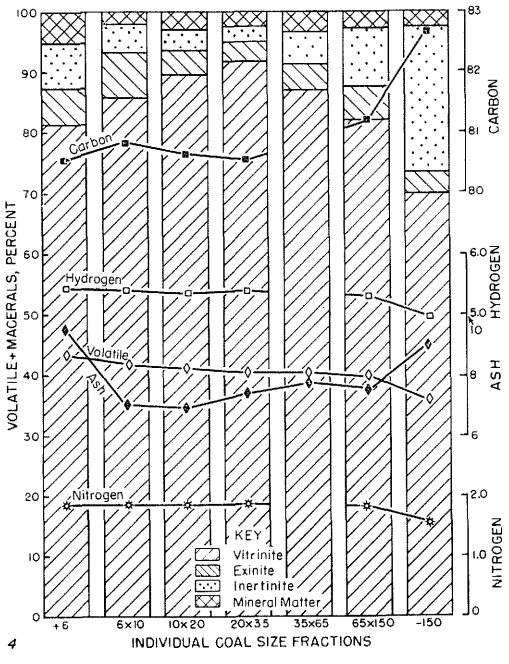
Macrotype or seam sample	Maceral proportions percent				Maceral (constituent) mean dimensions, microns				Maceral (constituent), quartile and median dimensions, microns											
									25% quartile				50% quartile (median)				75% quartile			
	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral
Reference pillar	86.7	5.8	5.0	2.5	40.1	3.7	11.2	6.1	4.9	1.6	2.5	2.2	13.0	2.6	5.2	4.1	41.7	5.0	10.6	7.0
Fine clarain	87.1	4.8	4.2	3.9	33.8	3.1	9.1	6.1	4.9	1.5	2.1	2.1	12.3	2.4	4.0	4.2	37.3	4.1	8.2	7.7
Medium clarain	84.1	4.4	6.7	4.7	38.4	3.3	15.8	7.6	4.9	1.5	2.4	1.9	12.8	2.4	5.2	3.4	40.7	4.4	12.5	6.9
Coarse clarain	84.5	3.3	7.7	4.5	50.9	3.7	20.0	8.0	5.3	1.6	2.9	2.6	14.9	2.7	5.8	5.0	47.4	5.0	12.1	8.4
High-vitrain clarain ("Vitrain")	91.1	1.3	4.3	3.2	89.2	3.0	32.1	9.2	7.0	1.5	2.3	2.9	24.8	2.2	4.9	5.2	85.3	3.3	12.4	9.5
Seam section I	88.6	3.5	5.5	2.4	60.1	4.0	25.4	6.2	6.2	1.7	2.4	2.4	20.7	2.8	5.3	4.6	64.8	5.6	12.2	7.7
Seam section II	81.1	5.2	7.6	6.2	40.0	4.3	20.6	9.8	4.8	1.8	2.7	2.8	12.6	3.1	5.6	5.1	40.4	5.8	12.7	8.8
Seam section III	83.7	5.3	9.3	1.7	36.5	3.4	14.2	6.7	5.0	1.5	2.2	2.2	12.9	2.4	4.3	4.1	39.1	4.2	9.2	7.1
Seam section IV	91.8	3.5	3.0	1.6	50.3	3.3	12.8	6.3	6.7	1.5	2.5	2.4	20.5	2.4	5.2	4.5	59.4	4.1	11.2	7.4
Representative sample	84.5	4.4	4.7	6.4	41.0	3.3	11.7	20.5	5.9	1.5	2.1	2.6	16.0	2.4	4.6	6.0	48.0	4.2	11.0	17.0
Fusain (-150 mesh)	2.1	.0	97.5	0.4																
Medium clarain (re-crushed at 1/32")	88.3	4.1	6.0	1.6	38.8	3.4	13.9	5.0	5.2	1.6	2.2	1.8	14.0	2.7	4.6	3.0	43.0	5.0	9.6	6.1

TABLE 3B.—No. 6 COAL: MICROMETRIC (PETROGRAPHIC) ANALYSES OF INDIVIDUAL SIZE FRACTIONS OF STANDARD SAMPLES

Sample size (Mesh)	Maceral proportions percent				Maceral (constituent) mean dimensions, microns				Maceral (constituent) quartile and median dimensions, microns											
									25% quartile				50% quartile (median)				75% quartile			
	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral
Reference Pillar																				
+6	81.7	5.7	7.5	5.1	46.9	4.0	13.1	9.8	4.4	1.6	2.5	3.2	10.2	2.5	5.2	5.4	42.0	4.8	10.0	8.7
6 x 10	85.6	7.5	4.7	2.2	33.2	3.6	8.9	4.3	4.1	1.6	2.6	2.1	9.6	2.6	5.2	3.9	31.0	5.0	9.5	6.6
10 x 20	89.6	4.0	3.4	3.0	50.3	3.8	15.0	8.2	5.8	1.5	2.1	2.4	17.0	2.4	4.7	4.5	54.0	4.6	11.5	7.4
20 x 35	91.6	3.5	2.5	2.4	57.1	3.5	11.7	7.1	5.8	1.6	2.4	2.3	19.0	2.6	5.5	4.4	63.0	4.8	13.5	7.8
35 x 65	87.0	4.1	5.6	3.3	43.4	4.2	14.4	10.3	6.1	1.7	3.3	1.8	17.0	2.9	5.5	3.4	54.0	5.6	8.9	7.7
65 x 150	82.2	5.5	9.7	2.6	37.8	4.7	15.4	9.3	6.2	1.9	3.5	2.6	18.5	3.5	6.4	4.8	53.0	6.1	16.0	7.9
−150	70.0	3.5	23.9	2.6	15.7	4.4	10.2	5.4	4.1	1.6	2.5	2.0	8.6	2.6	5.2	3.9	21.0	5.2	10.0	6.8
Fine Clarain																				
+6	79.15	10.65	5.86	4.34	31.34	5.39	12.06	9.54	6.2	2.1	2.8	3.6	19.0	4.0	6.0	6.1	50.0	6.8	12.5	9.6
6 x 10	88.01	5.21	3.20	3.58	36.93	3.09	9.32	6.27	4.4	1.55	1.9	2.1	11.0	2.4	3.5	4.2	37.0	4.2	7.4	8.0
10 x 20	86.30	4.77	3.70	5.23	30.61	2.92	7.48	5.96	5.7	1.5	2.1	2.1	15.0	2.2	4.2	4.1	41.0	3.7	8.1	7.5
20 x 35	86.05	4.04	6.20	3.71	34 03	3.20	10.45	5.64	4.6	1.5	2.4	2.0	12.0	2.3	4.8	3.9	33.0	3.8	9.0	7.0
35 x 65	87.69	4.08	4.65	3.57	31.95	3.48	9.80	5.79	5.5	1.6	2.2	1.9	14.0	2.5	4.6	3.7	40.0	4.7	9.0	6.9
65 x 150	88.01	4.42	5.59	1.98	27.32	3.58	13.73	6.10	5.9	1.6	3.16	2.8	14.0	2.5	6.2	4.8	36.0	4.8	15.0	7.8
−150	80.69	2.91	13.95	2.45	15.82	3.64	7.87	8.03	4.8	1.6	2.9	2.7	9.5	2.6	5.2	5.2	21.5	5.0	8.5	9.0
Medium Clarain																				
+6	88.64	5.88	4.86	0.61	54.72	4.25	16.93	6.47	6.5	1.7	3.5	3.4	17.0	2.9	6.4	5.2	49.0	5.7	14.0	7.8
6 x 10	87.18	4.74	4.06	4.02	40.81	3.08	13.63	8.02	4.9	1.5	2.2	1.8	14.0	2.3	4.9	3.1	45.0	4.0	12.0	6.4
10 x 20	77.75	4.39	10.99	6.87	40.30	3.53	24.25	6.48	4.7	1.6	3.0	1.9	10.5	2.6	6.0	3.7	34.0	5.0	16.0	7.6
20 x 35	85.51	4.21	5.78	4.50	38.42	3.42	13.76	8.60	4.9	1.6	2.1	1.9	13.0	2.6	4.4	3.4	43.0	4.8	10.0	6.7
35 x 65	86.07	4.24	5.19	4.50	33.00	3.27	10.90	8.17	5.1	1.5	2.2	1.9	14.0	2.4	4.5	3.6	44 0	4.1	9.1	7.2
65 x 150	86.71	2.89	6.92	3.48	30.87	3.43	12.22	7.98	6.1	1.6	2.8	2.1	16.0	2.6	6.2	3.8	43.0	5.0	15.0	7.0
−150	77.32	3.33	16.72	2.63	13.69	3.50	7.45	6.38	3.9	1.6	2.6	2.1	7.6	2.5	4.8	4.4	18.0	4.7	8.2	8.4
Coarse Clarain																				
+6	85.42	5.29	8.16	1.13	72.24	5.22	17.83	9.81	5.8	2.0	3.0	2.4	16.0	3.8	5.7	4.4	55.0	6.9	9.6	8.0
6 x 10	80.02	3.85	10.19	5.94	44.10	3.64	26.50	8.15	4.6	1.6	3.2	2.5	11.0	2.7	6.0	5.0	35.0	5.0	12.0	8.9
10 x 20	89.48	2.98	3.60	3.94	68.28	4.12	13.49	8.35	5.6	1.6	2.5	2.7	16.0	2.6	5.5	5.0	54.0	4.8	12.0	8.2
20 x 35	91.29	2.55	3.85	1.81	57.78	3.29	14.29	5.65	6.4	1.6	2.7	2.5	24.0	2.6	5.3	4.6	76.0	4.8	9.0	7.1
35 x 65	88.90	3.17	4.47	3.46	47.73	3.75	13.30	11.58	5.8	1.6	2.4	2.6	20.0	2.6	5.1	5.2	67.0	5.1	10.0	9.5
65 x 150	87.79	2.50	7.77	1.93	38.51	3.79	22.67	7.80	8.9	1.6	3.3	2.7	24.0	2.7	7.8	4.8	59.0	5.4	31.0	7.6
−150	75.76	1.65	19.58	3.01	17.48	3.77	8.81	6.30	4.6	1.8	3.3	2.9	9.7	3.2	5.8	4.9	23.0	5.7	9.5	7.5

High-Vitrain Clarain ("Vitrain")

+6	92.21	3.14	3.62	1.03	11.12	4.8	18.8	12.8	5.6	1.8	2.2	4.0	16.0	3.3	5.0	7.4	58.0	6.5	14.0	17.0
6 x 10	89.46	1.49	6.23	2.82	97.72	3.05	49.41	8.63	5.6	1.5	2.4	3.1	20.0	2.2	5.1	5.5	86.0	3.2	14.0	9.8
10 x 20	91.61	1.19	2.40	4.81	87.70	3.03	15.35	10.64	7.1	1.5	2.0	2.9	24.0	2.1	4.2	5.3	80.0	3.1	10.0	9.6
20 x 35	95.33	1.24	1.42	2.01	85.94	2.94	10.68	7.58	9.2	1.5	1.8	2.0	37.0	2.2	3.6	4.0	100.0	3.5	8.8	7.8
35 x 65	94.68	0.82	2.64	1.86	75.05	2.33	19.06	7.93	13.0	1.4	2.2	2.4	44.0	2.0	4.6	4.7	105.0	2.8	9.4	8.0
65 x 150	91.92	0.66	4.19	3.23	52.73	2.74	27.00	16.00	13.0	1.5	5.3	3.6	38.0	2.3	13.0	6.5	74.0	3.7	31.6	14.0
−150	90.12	1.08	5.46	3.34	18.39	3.23	7.92	8.75	5.0	1.7	2.6	2.3	11.0	2.7	5.2	4.8	24.0	5.1	9.1	8.9
Seam Section I																				
+6	91.19	1.54	2.69	4.58	72.53	3.10	11.62	7.31	6.8	1.5	2.1	2.0	22.0	2.1	4.6	4.2	69.0	3.1	10.0	8.1
6 x 10	88.89	3.20	6.26	1.65	77.14	4.36	36.36	6.13	7.0	1.7	2.4	2.6	26.0	3.0	5.4	4.8	81.0	5.9	14.0	7.8
10 x 20	88.09	4.56	3.43	3.92	42.27	3.59	11.00	6.31	4.8	1.6	2.4	2.6	13.0	2.6	5.0	4.8	41.0	5.1	8.8	8.1
20 x 35	90.76	2.94	4.34	1.96	61.97	4.15	24.50	6.23	6.1	1.7	2.6	2.1	20.0	2.9	5.6	4.2	73.0	5.5	12.0	7.9
35 x 65	87.42	3.50	6.16	2.92	41.98	3.91	20.85	7.29	6.4	1.7	2.3	2.1	20.0	2.8	5.2	4.2	58.0	5.6	14.0	7.6
65 x 150	91.32	2.21	4.19	2.28	36.33	3.40	12.76	5.41	7.6	1.6	2.7	1.8	22.0	2.6	5.8	3.4	53.0	4.9	13.0	6.5
−150	80.55	2.77	13.85	2.83	16.03	3.37	7.96	4.57	4.4	1.6	2.7	1.6	8.7	2.7	5.1	2.6	21.0	5.1	8.8	5.5
Seam Section II																				
+6	76.78	6.40	9.49	7.33	48.50	7.02	36.05	11.79	4.8	1.5	2.7	3.4	12.0	2.4	5.5	6.2	36.0	4.3	10.0	12.0
6 x 10	79.01	6.33	6.08	8.58	35.38	4.43	17.03	10.48	4.1	1.8	2.6	3.3	9.4	3.1	5.2	5.6	30.0	5.9	9.2	9.6
10 x 20	80.12	4.81	10.46	4.61	45.19	4.06	30.41	9.68	5.2	1.8	2.7	2.2	14.0	3.2	6.0	4.4	43.0	5.9	18.0	8.2
20 x 35	86.38	4.07	6.08	3.47	48.62	3.69	18.27	8.42	5.3	1.7	2.1	2.4	16.0	2.8	4.6	4.8	56.0	5.2	9.3	8.1
35 x 65	84.19	4.31	7.70	3.80	53.70	5.85	24.64	11.56	6.1	1.8	4.6	2.9	21.0	3.0	8.4	5.2	74.0	5.9	26.0	8.8
65 x 150	86.12	3.60	6.44	3.84	34.30	3.96	13.86	8.99	6.0	1.8	2.9	2.7	18.0	3.4	5.7	4.8	51.0	6.0	12.0	7.4
−150	81.16	2.89	12.05	3.90	15.85	3.88	6.89	5.73	4.5	1.8	2.7	2.1	9.0	3.2	5.0	4.2	22.0	5.8	8.0	7.2
Seam Section III																				
+6	78.38	5.69	8.82	7.11	38.74	4.21	11.21	9.07	4.6	1.8	3.1	3.3	9.8	3.1	5.5	5.9	34.0	5.9	9.4	11.0
6 x 10	86.70	5.64	6.67	.99	36.95	3.24	14.70	4.10	5.0	1.5	1.8	2.0	13.0	2.3	3.4	3.8	37.0	3.6	7.6	6.5
10 x 20	81.92	6.11	9.78	2.19	34.16	3.27	12.82	7.37	4.2	1.5	2.2	2.1	9.4	2.4	4.6	4.1	33.0	4.4	9.4	7.6
20 x 35	82.15	4.55	9.49	3.81	49.71	3.89	18.83	17.15	6.0	1.6	3.0	3.4	19.0	2.6	6.2	5.5	63.0	4.9	15.0	8.2
35 x 65	87.61	4.41	6.69	1.29	38.34	3.60	11.95	5.79	6.0	1.6	2.6	2.5	17.0	2.5	5.0	4.4	50.0	4.9	8.7	6.9
65 x 150	82.52	3.72	11.64	2.12	26.91	3.39	11.87	7.10	5.6	1.6	2.7	2.4	14.0	2.6	5.4	4.8	39.0	4.8	10.0	8.5
−150	58.75	2.65	36.97	1.62	14.73	3.84	8.91	4.31	4.2	1.6	3.5	1.8	8.1	3.0	6.0	3.3	19.5	5.5	10.0	6.2
Seam Section IV																				
+6	93.42	3.51	1.73	1.34	70.32	3.46	15.82	6.25	7.6	1.5	3.3	3.2	32.0	2.4	6.8	4.9	84.0	4.2	19.0	7.8
6 x 10	92.80	3.31	2.54	1.34	56.41	2.90	11.06	4.95	6.2	1.5	2.6	2.4	20.0	2.2	5.4	4.4	62.0	3.5	11.5	7.0
10 x 20	91.82	4.40	2.30	1.48	46.34	3.47	10.47	5.24	7.0	1.5	2.0	2.4	21.0	2.4	4.5	4.4	55.0	4.2	11.0	7.3
20 x 35	89.73	2.99	5.23	2.05	53.49	3.55	25.46	9.96	7.6	1.6	2.5	2.7	23.5	2.5	4.9	5.2	72.0	4.7	8.7	8.8
35 x 65	91.14	3.25	2.26	3.36	44.83	3.55	9.30	13.63	7.0	1.6	2.9	2.5	22.0	2.4	5.5	4.8	61.0	4.3	9.6	8.0
65 x 150	92.37	2.64	3.84	1.15	33.93	3.84	18.34	6.45	8.4	1.6	3.4	3.16	20.0	2.7	7.4	5.2	48.0	5.1	21.5	8.6
−150	87.29	2.98	8.06	1.67	16.17	4.68	6.60	3.77	4.4	1.8	2.3	1.6	8.9	3.2	4.5	2.4	21.0	6.2	7.8	4.4



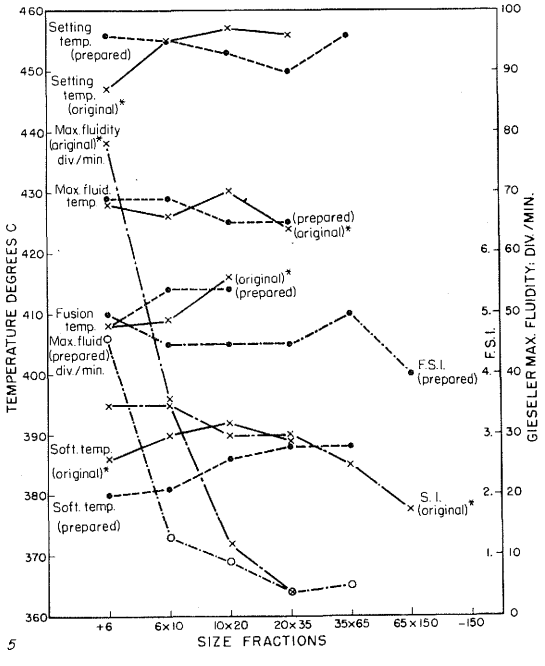


FIG. 4.—No. 6 coal: Petrographic constitution and chemical composition of individual size fractions of reference pillar, standard preparation (tables 3A and 5).

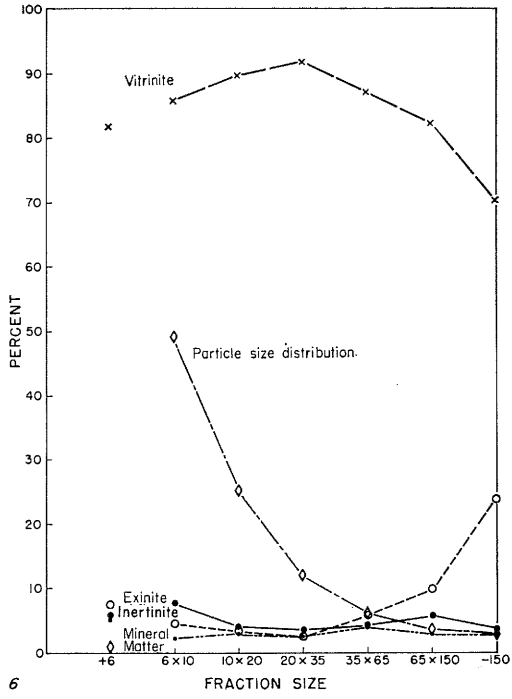
FIG. 5.—No. 6 coal: Gieseler values and free swelling indices for size fractions of reference pillar (standard preparation), also fluidity and swelling indices obtained from modified procedures (table 5). *See page 45.

the seam section samples are arranged simply in order of succession.

In the coal macro-types, as compared with that of the reference pillar, the proportions of the dominant constituent vitrinite were slightly reduced in the medium and coarse clarains; the high-vitrain clarain or "vitrain" featured an increase of approximately 3 percent. The macro-type series exhibits a systematic and progressive decrease in exinite content whereas the proportions of inertinite are conspicuously greater in both the medium and coarse clarains. The coarse, medium, and fine clarain have appreciably greater contents of microscopically identifiable mineral matter than the high-vitrain clarain.

The four seam sections show an interesting and significant distribution of constituent proportions (fig. 2). The content of the dominant constituent vitrinite is greater in both the lower (I) and upper (IV) sections of the seam; the two central sections (II and III) yield higher proportions of both exinite and inertinite. The amount of microscopically visible mineral matter is appreciably greater in seam section II than in the other three.

The volumetric proportions and constituent (maceral) size distributions in the various particle size ranges of the broken coal samples are of particular interest. It should be noted that marked variations which may be exhibited in the plus 6-mesh size may generally be a function of the small amount of this fraction which was a maximum of 0.31 percent of the sample.



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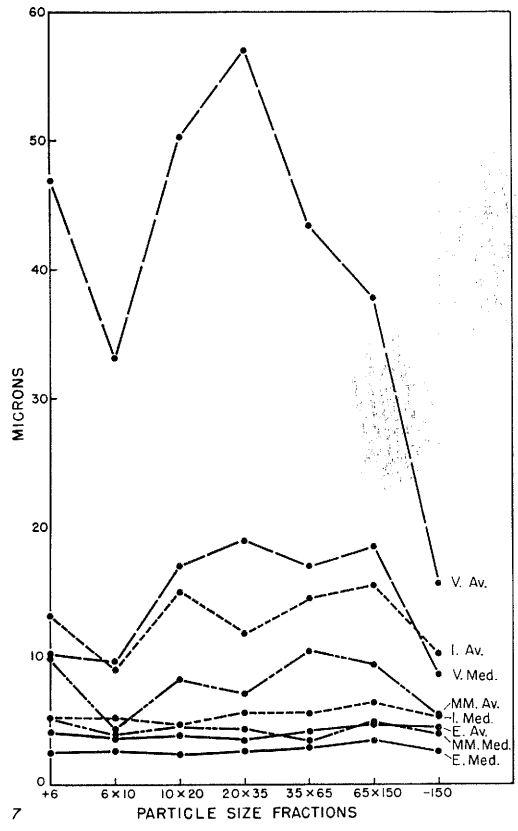

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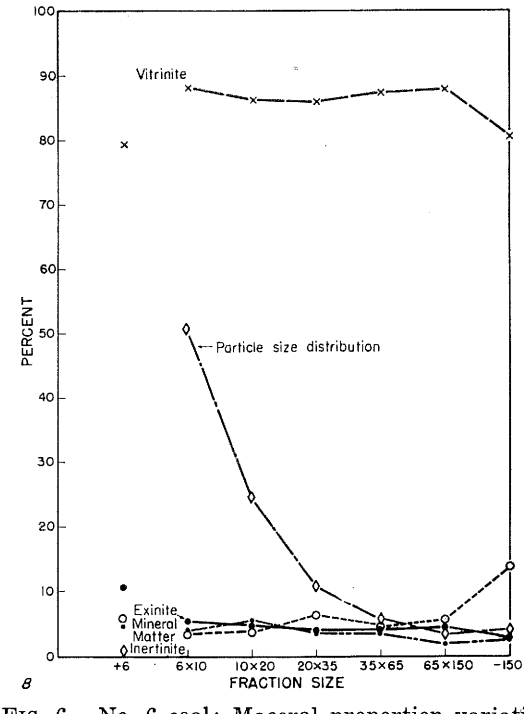

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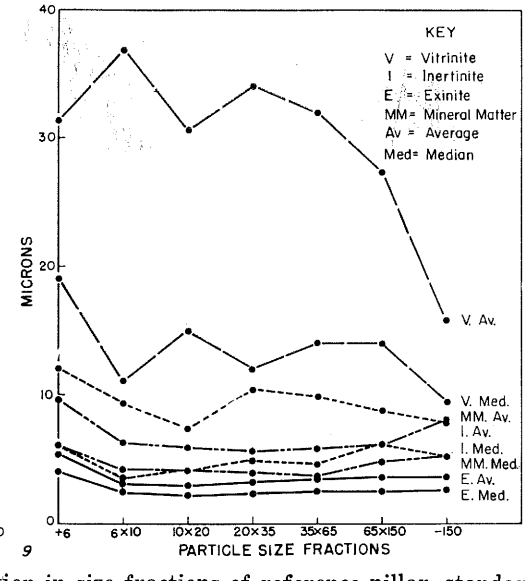

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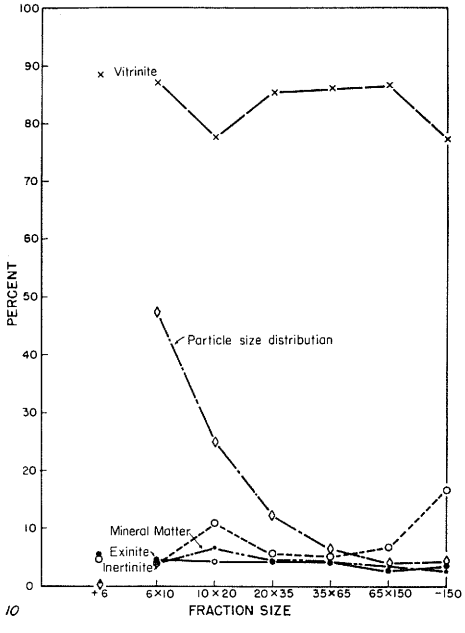
FIG. 6.—No. 6 coal: Maceral proportion variation in size fractions of reference pillar, standard preparation (tables 2 and 3B).

FIG. 7.—No. 6 coal: Maceral size variation in size fractions of reference pillar, standard preparation (table 3B).

FIG. 8.—No. 6 coal: Maceral proportion variation in size fractions of fine clarain, standard preparation (tables 2 and 3B).

FIG. 9.—No. 6 coal: Maceral size variation in size fractions of fine clarain, standard preparation (table 3B).



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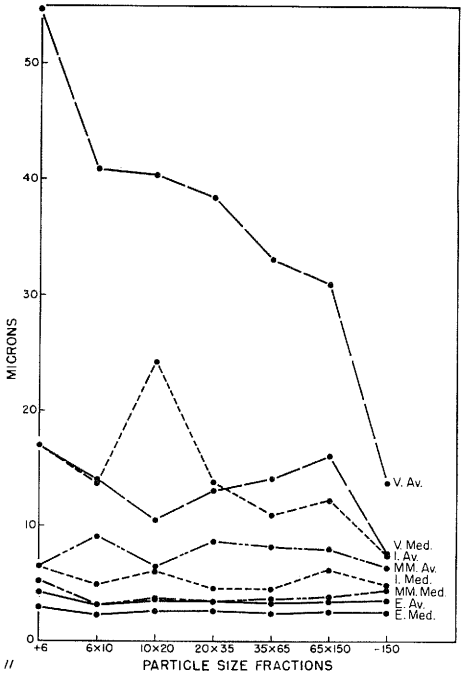

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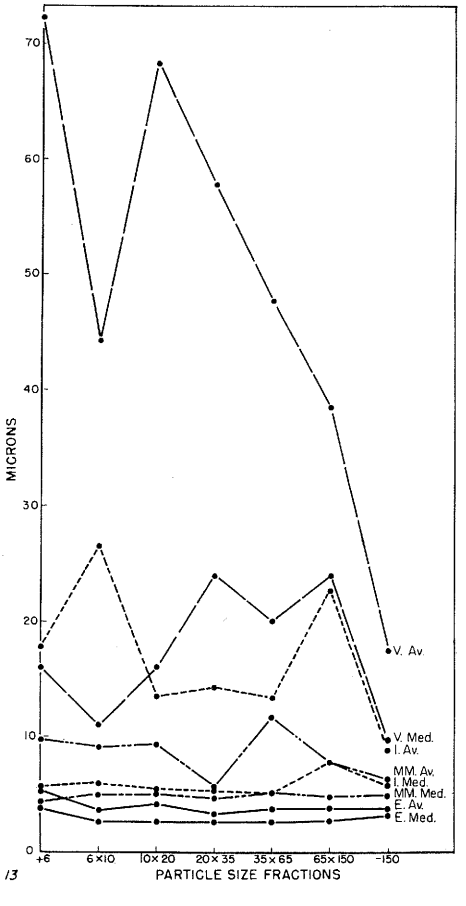

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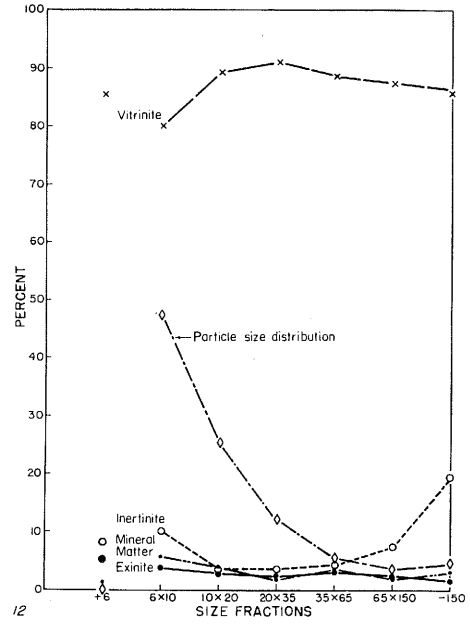

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KEY

V = Vitrinite
I = Inertinite
E = Exinite
MM = Mineral Matter
Av = Average
Med = Median

The particle size fractions of the reference pillar samples exhibit a progressive variation in the proportions of vitrinite from plus 6-mesh which reaches a maximum in the 20 x 35 mesh fraction and thereafter declines to a minimum value in the minus 150-mesh fraction (figs. 4 and 6). Conversely the content of inertinite declines from the plus 6-mesh to a minimum in the 20 x 35 mesh range and reaches a substantial maximum in the minus 150-mesh size fraction. The variation in the proportion of exinite shows two minima; one in the 20 x 65 mesh range and the other in the minus 150-mesh fraction. The mineral matter content is minor and variations are irregular.

The results of analysis of width of macerals and mineral matter in the particle size fractions of the representative pillar (table 3B, fig. 7) show an irregular but slight increase in median width of exinite and inertinite up to the 65 x 150 mesh size after the decline from the very small amount of the plus 6-mesh fraction. In all components except vitrinite a decrease in the average width in the 20 x 35 mesh size is shown; vitrinite reaches a maximum average width in this size range. In all cases except for the average width of exinite, which remains about the same, there is a decrease in average and median width of the macerals in the minus 150-mesh size. In general, the range of variations within the median widths was not as great as in the average widths.

The coal constituent (maceral) proportions of the fine clarain exhibit a very similar general trend to that of the pillar sample, but with two less prominent maxima in vitrinite content in the 6 x 10 and 65 x 150 particle size ranges (table 3B, fig. 8). Variations in the proportions of inertinite, exinite, and mineral matter are similar to those of the reference pillar, especially the well defined maximum content of inertinite in the minus 150-mesh range.

Variation in the *sizes* of the coal constituents (macerals) throughout the particle size fractions demonstrates the initial breakdown of the relatively few grosser bodies of vitrinite and fusinite (recorded as inertinite) as indicated in the average and median constituent size values (fig. 9). Thereafter the median sizes of both these constituents increases in the progressively finer particle size ranges. After an initial decrease in median size through the 10 x 20 mesh fraction, this tendency is present but to a lesser degree in both average and median size values for the exinite and mineral matter.

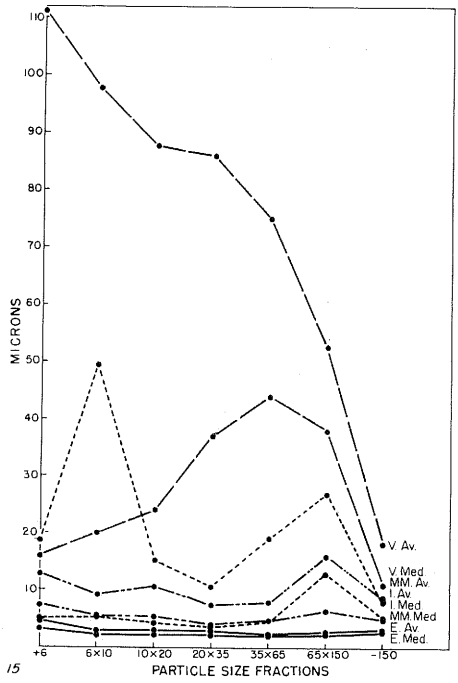
The medium, coarse, and high-vitrain clarains, after an initial general decline in the vitrinite proportions of the plus 6 and 6 x 10 mesh ranges (also the 10 x 20 mesh of the medium clarain), exhibit an increase in the proportions of this constituent in the middle particle size ranges followed by a progressive decline to the minus 150-mesh fraction; with the exception of the medium clarain (in which it is barely so), this is not the minimum value for the particle size fractions of their respective size series (figs. 10, 12, and 14).

FIG. 10.—No. 6 coal: Maceral proportion variation in size fraction of medium clarain, standard preparation (tables 2 and 3B).

FIG. 11.—No. 6 coal: Maceral size variation in size fractions of medium clarain, standard preparation (table 3B).

FIG. 12.—No. 6 coal: Maceral proportion variation in size fractions of coarse clarain, standard preparation (tables 2 and 3B).

FIG. 13.—No. 6 coal: Maceral size variation in size fractions of coarse clarain, standard preparation (table 3B).



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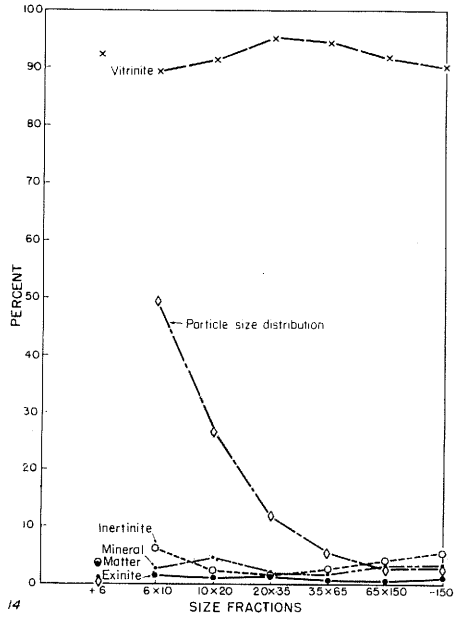

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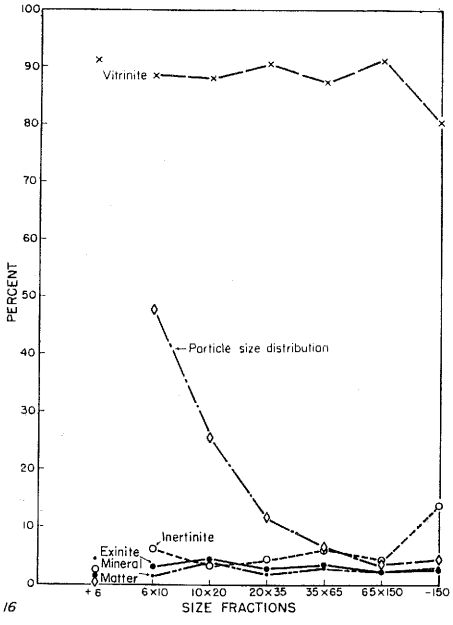
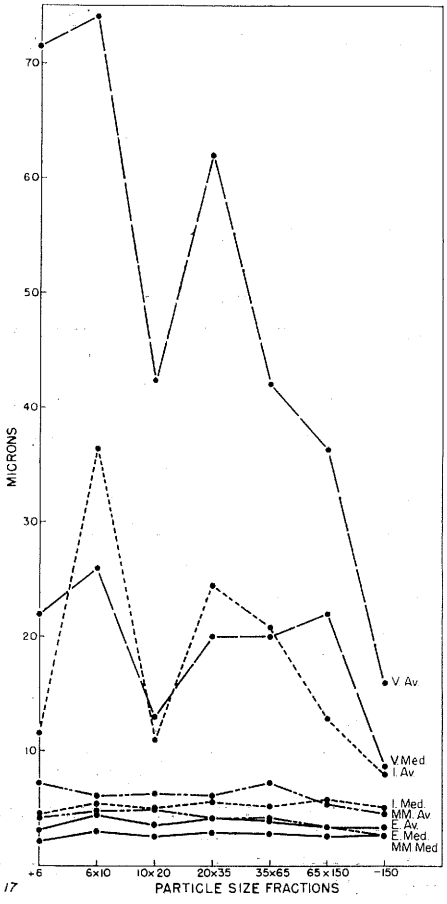


16

KEY

V = Vitrinite
I = Inertinite
E = Exinite
MM = Mineral Matter
Av = Average
Med = Median



17

In these coal macro-types (medium, coarse, and high-vitrain clarain) the variations in constituent (maceral) sizes exhibit significant and progressive changes (figs. 11, 13, and 15). With the exception of one particle size range fraction in the coarse clarain (6 x 10 mesh), the *average* constituent size for vitrinite decreases rapidly in each of the respective particle size series from plus 6 to minus 150-mesh. In respect of *median* constituent size, after an initial size decrease from plus 6 to 6 x 10 mesh fractions in the case of medium and coarse clarain, there is a well defined *increase* in size, which persists generally into the 35 x 65 or 65 x 150 mesh range; in all cases there is a prominent median size reduction in the minus 150-mesh size group.

The maceral size variation of the inertinite is rather more erratic, generally exhibiting major and subordinate maxima in both average and median values about the 6 x 10 or 10 x 20 mesh ranges, and the 65 x 150 mesh size respectively. After an initial slight decline in median size from the plus 6-mesh fraction to the 6 x 10 mesh fraction, the average and median maceral sizes for exinite exhibit little variation.

In these macro-type samples, there is sympathetic variation between constituent proportions and constituent median sizes in respect to vitrinite (the dominant maceral) and to a lesser extent inertinite which includes fusain. These are the two constituents that consistently exhibit the greatest variation in sheet thickness and commonly the greatest individual dimensions. The minor and finer seam constituents, exinite and mineral matter, display much less variation.

These characteristics of the distribution of maceral proportions, average and median sizes, are of particular interest and potential importance in both preparation and utilization of coal.

As is evident under the microscope and demonstrated by comparison of particle (screen) and constituent sizes throughout the size fraction range, the great majority of the coal particles are of composite character.

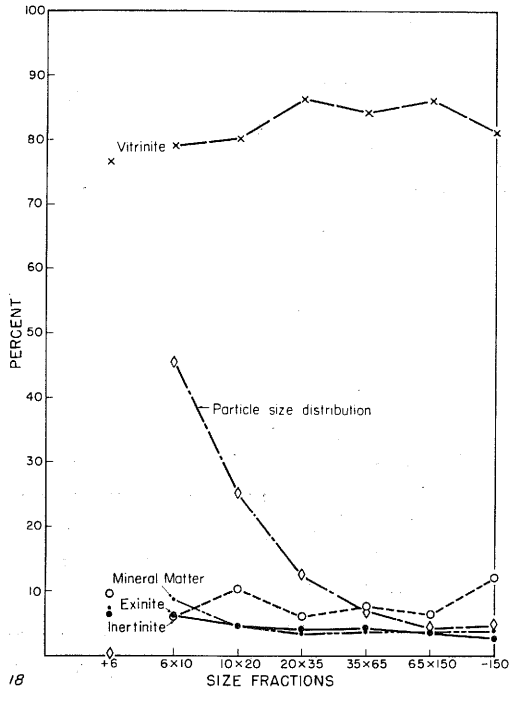
From a consideration of the maceral proportions and sizes in the various fractions of the broken coal, it would thus appear that the seam components or coal types most resistant to mechanical degradation are the finer banded clarains, of which more finely divided vitrinite and fusinite are characteristic. These coal types with their more finely comminuted plant debris (macerals) would therefore not appear so prominently in the finer particle size ranges of the broken coal. However, both seam character and breakage conditions would introduce variation. The brittleness, closely developed and fine jointing characteristic of the coarser vitrain bands, and the lower mechanical strength exhibited by the larger and relatively mineral-free fusain lenses, predisposes these macerals to mechanical disintegration.

FIG. 14.—No. 6 coal: Maceral proportion variation in size fraction of "vitrain" (high-vitrain clarain), standard preparation (tables 2 and 3B).

FIG. 15.—No. 6 coal: Maceral size variation in size fractions of "vitrain" (high-vitrain clarain), standard preparation (table 3B).

FIG. 16.—No. 6 coal: Maceral proportion variation in size fraction of seam section I, standard preparation (tables 2 and 3B).

FIG. 17.—No. 6 coal: Maceral size variation in size fractions of seam section I, standard preparation (table 3B).



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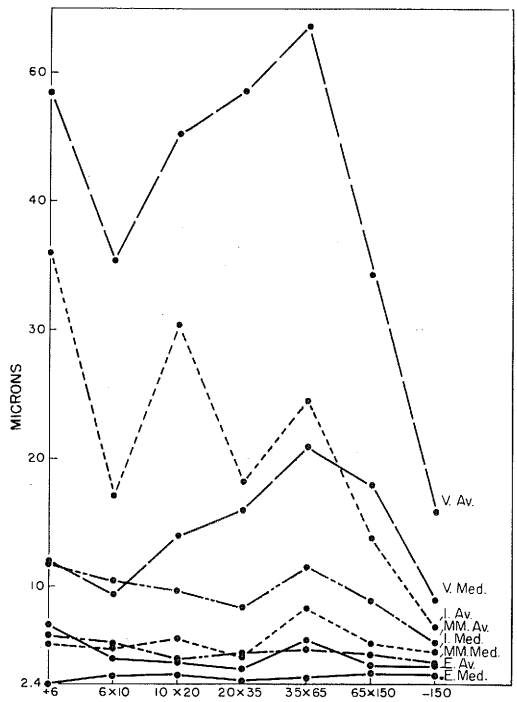

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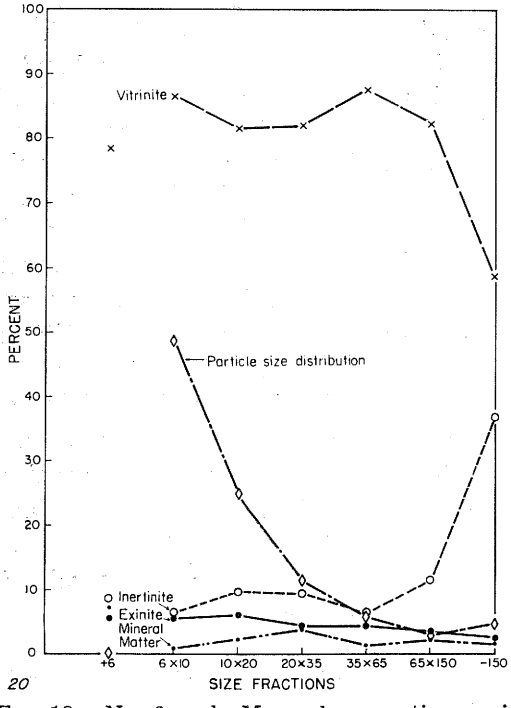

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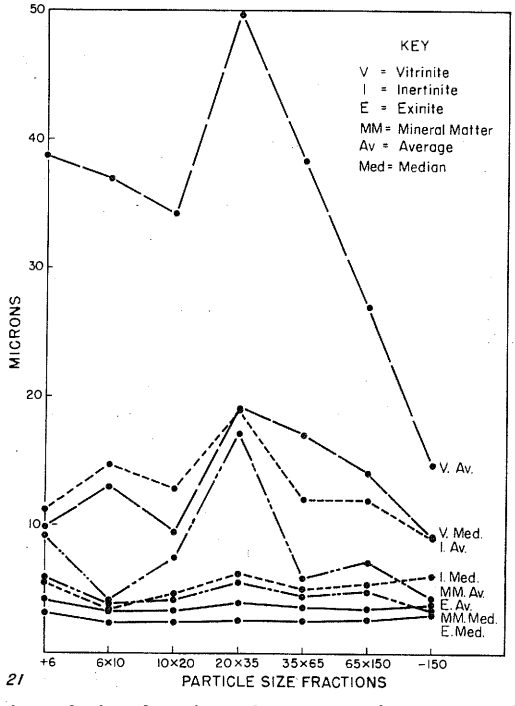

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FIG. 18.—No. 6 coal: Maceral proportion variation of size fraction of seam section II, standard preparation (tables 2 and 3B).

FIG. 19.—No. 6 coal: Maceral size variation in size fractions of seam section II, standard preparation (table 3B).

FIG. 20.—No. 6 coal: Maceral proportion variation in size fraction of seam section III, standard preparation (tables 2 and 3B).

FIG. 21.—No. 6 coal: Maceral size variation in size fractions of seam section III, standard preparation (table 3A).

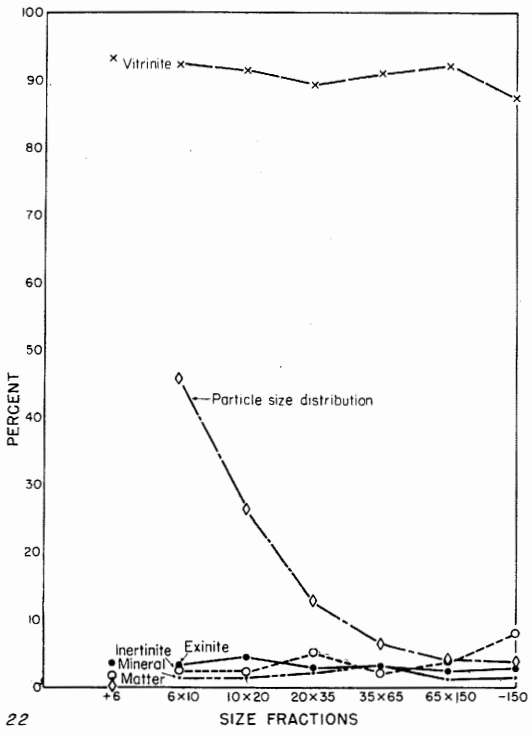


FIG. 22.—No. 6 coal: Maceral proportion variation in size fractions of seam section IV, standard preparation (tables 2 and 3B).

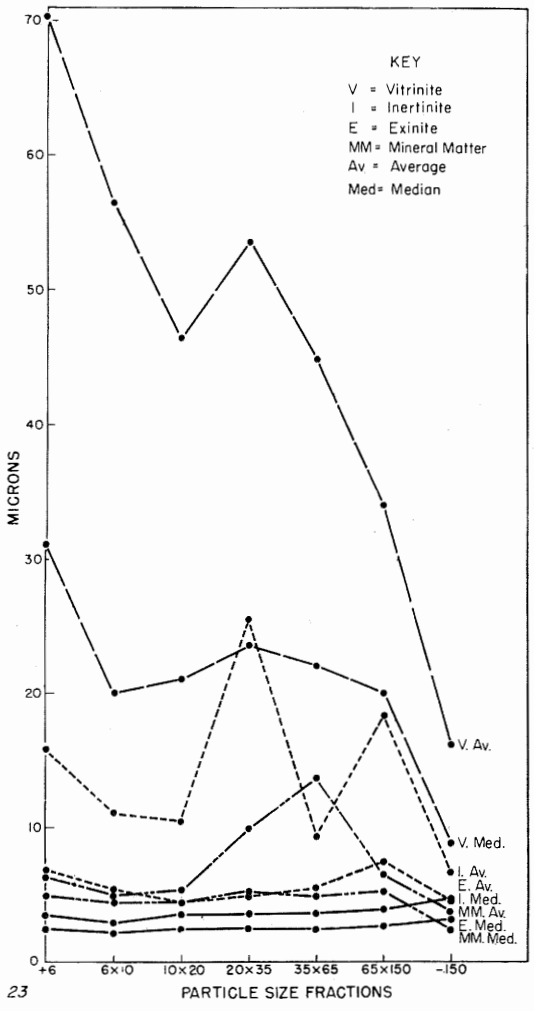
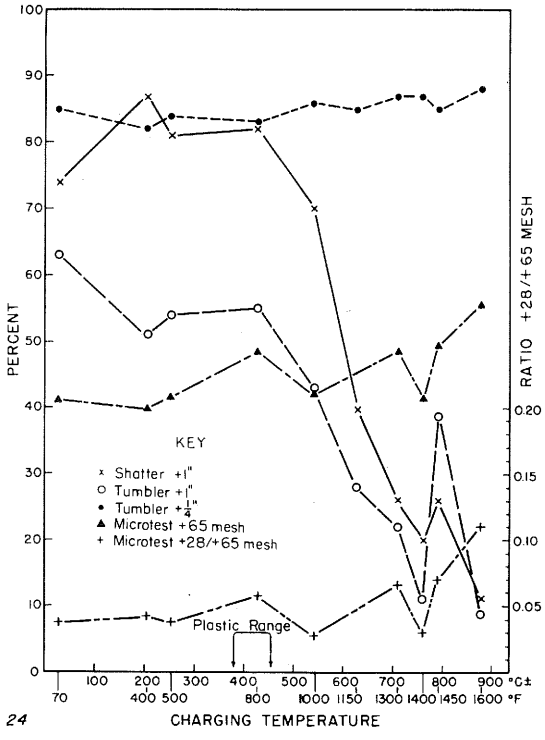


FIG. 23.—No. 6 coal: Maceral size variation in size fractions of seam section IV, standard preparation (table 3B).

Petrographic study of the particle size fractions of the seam sections (figs. 16 to 23) revealed trends generally comparable with those of the macrotypes described above. The lower (SS I) and upper (SS IV) seam sections, both with higher proportions of vitrinite than the middle sections, displayed in their particle size fractions, a slightly erratic but generally decreasing proportion of this maceral from the coarser to the finer grades (figs. 16, 18, 20, and 22). Vitrinite generally increased in proportion with decrease of particle size in fractions of seam section II with a decrease in the minus 150-mesh fraction. Seam section III exhibited maxima in vitrinite content in the 6 x 10 and 35 x 65 mesh ranges with a precipitous decline in proportion of vitrinite in the minus 150-mesh fraction (fig. 20). In all seam sections the inertinite content of the fractions generally varied antithetically with that of vitrinite. In seam sections II and III the exinite proportions decreased generally in the successively finer size ranges (figs. 18 and 20).

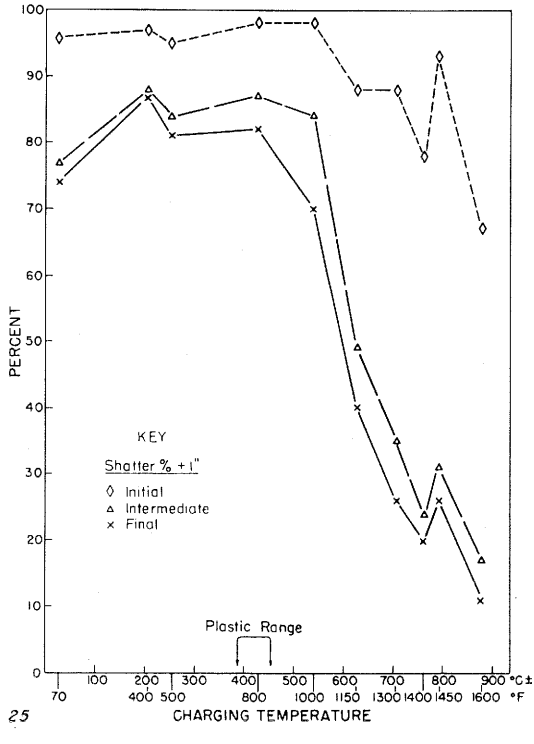


24

FIG. 24.—No. 6 coal: Influence of charging temperature on coke. Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period 2 hours (table 6).

FIG. 25.—No. 6 coal: Influence of charging temperature on coke—progressive degradation in shatter test. Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period 2 hours (table 6).

FIG. 26.—No. 6 coal: Influence of charging temperature on coke: progressive degradation in tumbler test. Reference pillar, standard size consist, rate of temperature increase, final coking temperature and final coking period 2 hours (table 6).



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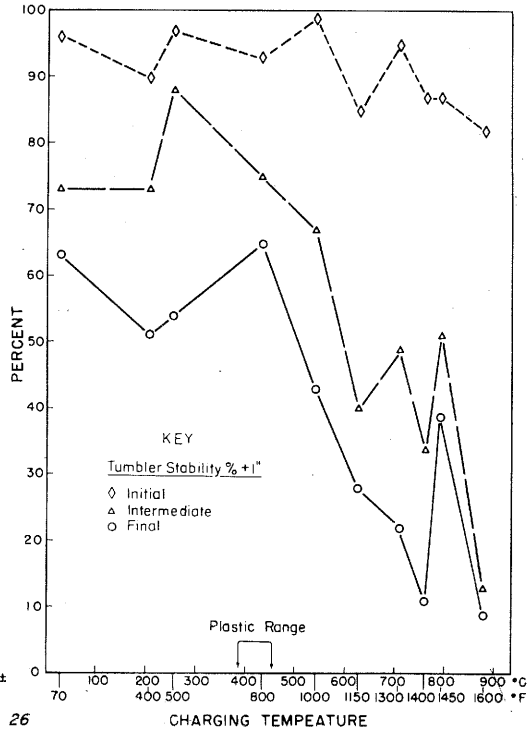

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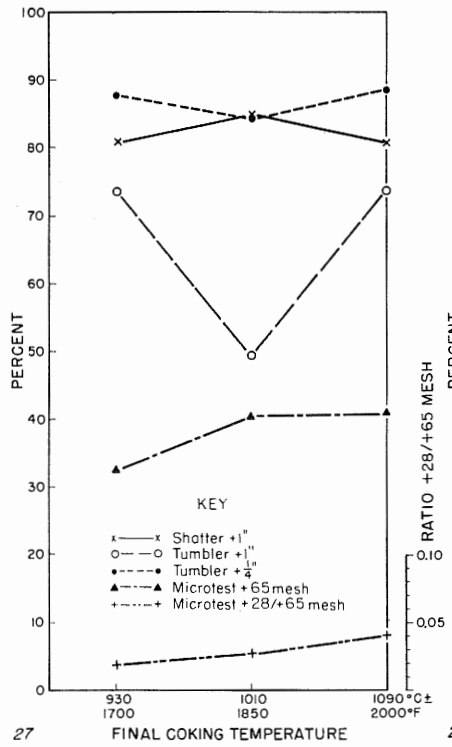
As each seam section contained bands representative of the various coal types, the average and median size trends of the macerals, although present, are neither so clearly defined nor so consistent in behavior as in the individual macrotype samples. In seam sections I and IV (lowest and uppermost and containing the highest vitrinite over-all proportion), the size maxima occur in the coarsest particle grades (figs. 17 and 23). Subordinate maxima appear in the 20 x 35 mesh or smaller particle size ranges, followed by a sharp median size decline to the finest fraction. In the two middle seam sections (II and III), after some initial fluctuation, both average and median sizes of the vitrinite rise to a maximum in the 35 x 65 and the 20 x 35 mesh particle size fractions respectively, and thence decline with decreasing particle size (figs. 19 and 21). In a general way, the average and median sizes for inertinite exhibit similar trends.

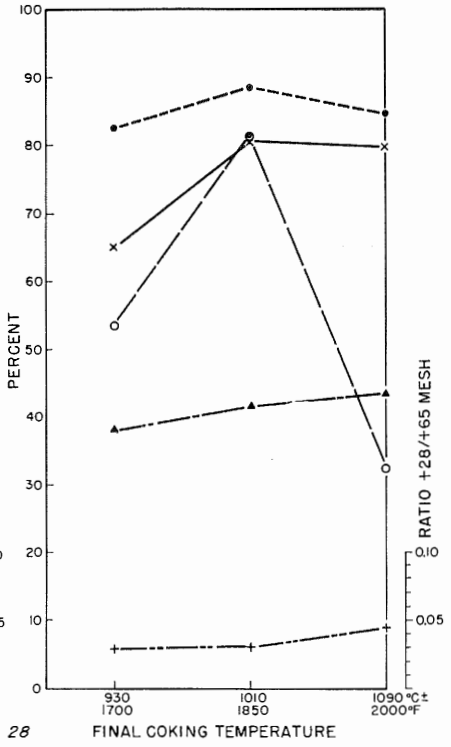
Size distribution characteristics of the mineral matter show a broad correlation with those of vitrain and inertinite. Those of exinite display a much greater uniformity in a general and gentle increase of median size from the coarsest to the finest particle size groups in seam sections II, III and IV. In seam sections III and IV there is a slight initial decline to the 6 x 10 mesh particle size range; in seam section I the median size of the exinite varies but slightly after a small initial increase.

These petrographic studies of volumetric proportions and size distributions of the more important macerals in the coal types and seam sections emphasize the nature of the variation which might be induced in the characteristics of prepared coal by modification of methods of breakage and selection of size ranges. It would appear that, under the circumstances of preparation adopted in this investigation, the various particle size fractions are characterized by marked variation in distribution of the proportions and sizes of the different macerals. It is evident that any departure from uniformity in respect to particle size distribution in the oven charge must result in variations of proportions, size characteristics, and distribution of the coal constituents and types, with probable consequent modification of the coking characteristics.

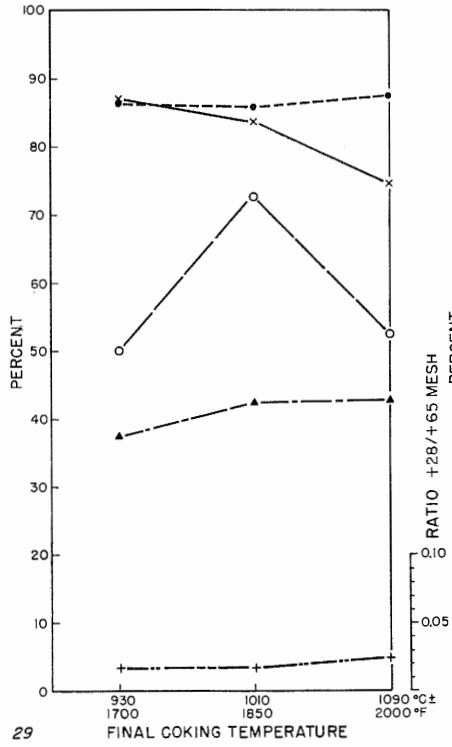
Similarly, intentional or accidental variation of seam representation in the oven charge, either through selective mining or other causes, might have appreciable effects upon the quality of the coke produced. More information is needed about the relation of size of broken coal produced under different conditions of preparation and the petrographic constitution of such coal so as to provide a sound basis for directing preparation of material of preferred size and petrographic character.


PERCENT
RATIO +28/+65 MESH
KEY
x——x Shatter +1"
o— —o Tumbler +1"
•----• Tumbler +¼"
▲—·—▲ Microtest +65 mesh
+—··—+ Microtest +28/+65 mesh
930 1700
1010 1850
1090 °C± 2000°F
FINAL COKING TEMPERATURE

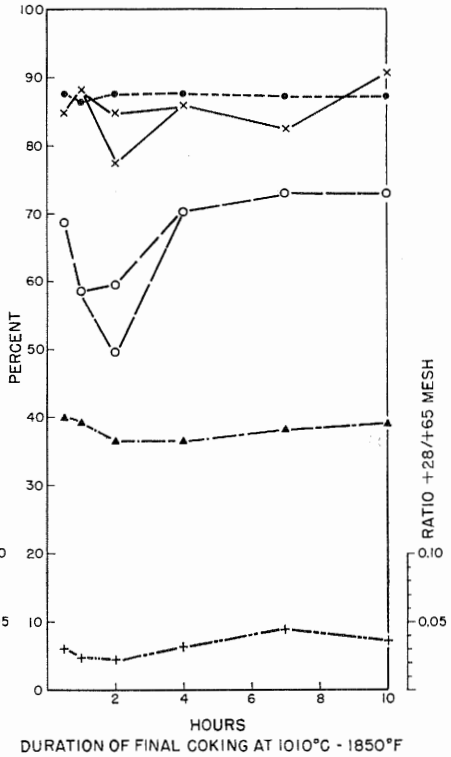
27


PERCENT
RATIO +28/+65 MESH
930 1700
1010 1850
1090 °C± 2000°F
FINAL COKING TEMPERATURE

28


PERCENT
RATIO +28/+65 MESH
930 1700
1010 1850
1090 °C± 2000°F
FINAL COKING TEMPERATURE

29


PERCENT
RATIO +28/+65 MESH
HOURS
DURATION OF FINAL COKING AT 1010°C - 1850°F

30

FIG. 27.—No. 6 coal: Influence of final coking temperature on coke. Reference pillar, standard size consist, standard charging temperature, rate of temperature increase, and final coking period 2 hours (table 8).

FIG. 28.—No. 6 coal: Influence of final coking temperature on coke. Medium clarain, standard size consist, standard charging temperature, rate of temperature increase, and final coking period 2 hours (table 8).

FIG. 29.—No. 6 coal: Influence of final coking temperature on coke. Coarse clarain, standard size consist, standard charging temperature, rate of temperature increase, and final coking period 2 hours (table 8).

FIG. 30.—No. 6 coal: Influence of final coking period on coke. Reference pillar, standard size consist, charging temperature, rate of temperature increase, and final coking temperature (table 9).

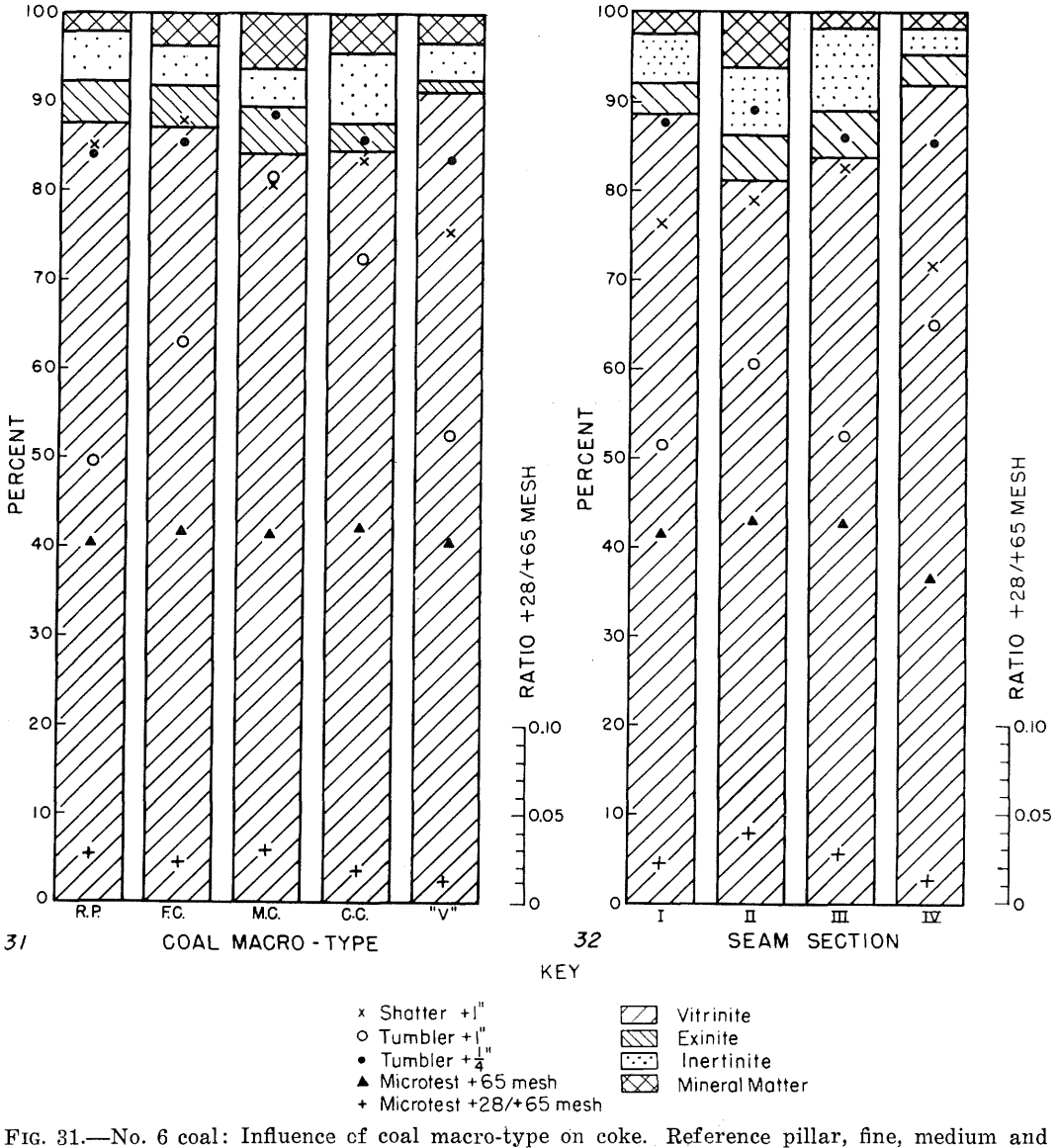


FIG. 31.—No. 6 coal: Influence of coal macro-type on coke. Reference pillar, fine, medium and coarse clarains, "vitrain" all in standard size consists, and standard coking conditions (tables 3A, 10).

FIG. 32.—No. 6 coal: Influence of seam sections on coke. Standard size consist and standard coking conditions (tables 3A and 10).

TABLE 4.—No. 6 COAL: CHEMICAL ANALYSES, GIESELER PLASTICITY, AND FREE SWELLING INDEX
(Reference pillar, coal type, and seam section samples)

Sample, fraction, and/or condition	As Received				Moisture and ash free								Gieseler Plasticity data					Free swelling index
	Proximate				Proximate		Ultimate						Soft. temp. °C	Fusion temp. °C	Max. fluid temp. °C	Setting temp. °C	Max. fluid div/min.	
	Moist.	Ash	Vol.	F. C.	Vol.	F. C.	H	C	N	O	S	Btu/lb.						
Reference pillar																		
Entire	7.8	8.6	34.8	48.8	41.6	58.4	5.46	80.82	1.94	—	—	14538	385	413	425	456	15	4
+6 mesh	8.5	9.0	35.1	47.4	42.5	57.5	5.56	81.00	1.93	9.76	1.75	14505	383	407	426	455	41	4
−6 mesh	9.1	7.3	34.8	48.8	41.6	58.4	5.52	81.57	1.93	9.31	1.67	14545	382	407	425	456	32	3½
Fine clarain																		
+6 mesh	6.4	9.3	36.6	47.7	43.4	56.6	5.51	80.14	1.96	9.98	2.41	14559	384	410	428	458	41	5½
−6 mesh	8.0	9.2	35.0	47.8	42.3	57.7	5.44	80.62	1.96	9.71	2.27	14528	383	409	428	454	32	5
Medium clarain																		
+6 mesh	8.9	10.4	34.7	46.0	42.9	57.1	5.62	81.30	1.90	9.59	1.59	14532	384	408	429	457	63	4½
−6 mesh	9.1	9.0	34.6	47.3	42.2	57.8	5.54	81.54	1.95	9.27	1.70	14500	382	407	427	456	37	4½
Coarse clarain																		
+6 mesh	9.8	9.4	33.2	47.6	41.1	58.9	5.49	81.68	1.89	9.78	1.16	14491	384	409	426	456	21	5½
−6 mesh	9.7	7.4	34.1	48.8	41.1	58.9	5.47	81.52	1.79	9.97	1.25	14461	382	407	422	455	24	3½
"High vitrain clarain"																		
+6 mesh	9.0	7.1	34.0	49.9	40.6	59.4	5.41	81.32	1.89	10.07	1.31	14523	386	412	426	458	20	4½
−6 mesh	9.5	5.9	33.6	51.0	39.7	60.3	5.34	81.28	1.93	10.19	1.26	14485	386	410	428	458	16	4½
Seam section I																		
+6 mesh	9.2	9.0	34.0	47.8	41.5	58.5	5.45	80.85	1.96	10.21	1.53	14494	382	407	426	451	29	4½
−6 mesh	8.8	8.5	34.2	48.5	41.4	58.6	5.42	80.88	1.98	10.06	1.66	14427	379	407	426	453	29	5
Seam section II																		
+6 mesh	9.2	12.4	32.0	46.4	40.8	59.2	5.54	81.23	1.89	10.34	1.00	14402	385	412	427	455	16	5
−6 mesh	9.6	10.1	32.1	48.2	39.9	60.1	5.38	80.91	1.92	10.79	1.00	14430	385	413	427	453	13	4½
Seam section III																		
+6 mesh	9.0	7.6	35.1	48.3	42.1	57.9	5.62	81.34	1.92	9.68	1.44	14454	384	408	429	460	61	4½
−6 mesh	9.5	6.1	34.8	49.6	41.2	58.8	5.43	81.50	1.92	9.72	1.43	14511	382	406	428	458	61	4½
Seam section IV																		
+6 mesh	8.7	5.8	34.7	50.8	40.6	59.4	5.52	80.47	1.82	9.83	2.36	14504	381	408	426	460	41	4
−6 mesh	8.8	5.4	36.2	49.6	42.2	57.8	5.44	80.33	1.80	10.11	2.32	14560	376	404	423	459	62	4

As a further measure of the character of the variations in the reference pillar, macro-type and seam section samples, proximate and ultimate analyses, Gieseler and free swelling index determinations were made upon the entire minus 6-mesh material (comprising approximately 99.8 percent of the sample) and the subordinate plus 6-mesh fraction. Each of the size fractions of the reference pillar was investigated in a similar manner.

Almost without exception, in the seam and macro-type samples, the minor plus 6-mesh fraction, as compared with the entire minus 6-mesh portion of the prepared material, was characterized by higher contents of ash, volatile matter, and hydrogen (table 4). The Gieseler values differed little in magnitude but, with few exceptions, those of the plus 6-mesh fraction were slightly higher than those of the minus 6-mesh portion, as were also the free swelling indices.

The variations in the principal values of the proximate, ultimate, and Gieseler determinations for the greatly dominant entire minus 6-mesh fractions of the seam and macro-type samples (table 4) are represented in figures 2 and 3 in association with the petrographic variations.

In the macro-type samples fixed carbon shows a very slight tendency towards antithetic variation with the vitrinite content and sympathetic relationship with the inertinite. Volatiles show a systematic and progressive variation with a very slight maximum in fine clarain in which sulfur is also slightly above normal; the content of the latter element is less in both coarse and high-vitrain clarains.

As regards the four seam sections (table 4, fig. 2), the greatly increased proportion of microscopically visible mineral matter in seam section II is reflected in a higher ash content. The sulfur content increases progressively in seam sections II, III and IV but is higher in section I than in sections II and III. Neither carbon nor hydrogen show any appreciably significant variation (possibly due to "cancellation" of the effects of increased exinite and inertinite in the middle seam sections); the slight variation in volatile content may be tentatively correlated with the more decisive changes in vitrinite content. Both the lower and upper sections of the seam feature increased proportions of total sulfur which are not reflected in the ash or mineral variation.

The principal values of the proximate and ultimate analyses of the various size fractions of the reference pillar sample as prepared under standard conditions appear in table 5 and are plotted against size and compared with the petrographic constitution in figure 4 (table 3B). There is a generally sympathetic variation between carbon and inertinite. Both hydrogen and volatile content decrease gradually and progressively from the coarser to the finer fractions, with a more marked decline in the last stage, probably to be associated with the integrated effects of diminishing vitrinite and exinite as well as greatly increased inertinite.

The Gieseler values and free swelling indices were determined upon each size fraction in accordance with accepted procedure for these tests. An additional fluidity determination was made using the same equipment and conditions used for Gieseler determinations except that the sample was not prepared to standard size (minus 40-mesh) but used in the testing as obtained from the sieved fractions (orig-

TABLE 5.—No. 6 COAL: CHEMICAL ANALYSES AND FLUIDITY AND SWELLING DATA OF REFERENCE PILLAR SAMPLE AND SIZE FRACTIONS
(Produced by standard procedure)

Sample, fraction, and/or condition (Mesh)	As received				Moisture and ash free												
	Proximate				Proximate		Ultimate						Plasticity data					
	Moist.	Ash	Vol.	F.C.	Vol.	F.C.	H	C	N	O	S	Btu/lb.	Soft. temp. °C	Fusion temp. °C	Max. fluid. temp. °C	Setting temp. °C	Max. fluid div/min.	Swelling index
Reference pillar	7.8	8.6	34.8	48.8	41.6	58.4	5.46	80.82	1.94	—	—	14538	385	413	425	456	15	4
+6*	8.5	9.0	35.1	47.4	42.5	57.5	5.56	81.00	1.93	9.76	1.75	14505	383	407	426	455	41	4
−6*	9.1	7.3	34.8	48.8	41.6	58.4	5.52	81.57	1.93	9.31	1.67	14545	382	407	425	456	32	3½
+6*	7.0	9.5	36.0	47.5	43.1	56.9	5.43	80.52	1.88	10.30	1.87	14405	380	408	429	456	46	5
+6†													386	408	428	447	78	3½
6 x 10*	8.2	7.0	35.2	49.6	41.5	58.5	5.41	80.87	1.84	10.25	1.63	14425	381	414	429	455	13	4½
6 x 10†													390	409	426	455	36	3½
10 x 20*	8.5	6.9	35.0	49.6	41.4	58.6	5.34	80.67	1.86	10.54	1.59	14459	386	413	425	453	9	4½
10 x 20†													392	416	430	457	12	3
20 x 35*	7.7	7.4	34.3	50.6	40.4	59.6	5.40	80.58	1.83	10.57	1.62	14432	388	—	425	450	4	4½
20 x 35†													389	—	424	456	4	3
35 x 65*	6.7	7.7	34.7	50.9	40.5	59.5	—	—	—	—	—	14313	388	—	425	456	5	5
35 x 65†													—	—	—	—	—	2½
65 x 150†	7.2	7.5	33.7	51.6	39.5	60 5	5 31	81.20	1.80	9.99	1.70	14059	—	—	—	—	—	4
−150†	4.4	9.0	31.0	55.6	35.8	64 2	4 95	82.64	1.57	9.12	1.72	14229	—	—	—	—	—	1¾

*Prepared—Gieseler plasticity data and free swelling index.
†Original—Fluidity and swelling data obtained by modified procedure.

inal size). Free swelling indices also were determined using accepted procedures except for using samples in the "original" size obtained by sieving instead of preparation of size according to the accepted procedures (minus 60-mesh) (table 5, fig. 5).

It was anticipated that any differences between these two sets of values would diminish as the original fraction size approached that of the accepted test specification. In each case the softening temperature proved to be slightly higher for the size fraction with its original size distribution as also were the setting temperatures with one exception; fusion and maximum fluidity temperatures varied more erratically. In the original samples (modified procedure) maximum fluidity was higher than in those prepared according to the accepted test specifications. The free swelling index was greater in all the prepared (i.e. accepted test) specimens.

Comparison of the fluidity values and free swelling indices of the modified procedure (samples of original size) for the various size fractions of the standard reference pillar (fig. 5) revealed an initial increase in both softening and setting temperatures with apparent maxima in the 10 x 20 mesh size fraction; unfortunately the series could not be completed. Maximum fluidity values (dial divisions per minute) declined from the plus 6-mesh fraction to the 20 x 35 mesh in both procedures.

The free swelling index was also decreased from the coarse to the finer fractions, particularly in samples in the modified test. The behavior of these specimens as between their original and standard prepared conditions appears to be anomalous even when considered in relation to variations of petrographic composition, maceral size, and probable distribution. Although present in relatively minor proportions, it is possible that the general decrease of exinite content in the finer size fractions is significant.

Coking Studies

Factors in the Coking Cycle

Broadly, the properties of coke have been considered as affected significantly by inherent and naturally induced characteristics of the seam (type and rank), by preparation procedures, and by the conditions of coking.

Limitations of equipment helped to establish certain arbitrary standards. Evaluation of the effects of various factors involved in the heating cycle constituted an essential introduction to all coking studies; without this information, it was not possible to define acceptable standard or optimum conditions of coking. Rates of temperature increase below, within, and above the plastic temperature range of the coal were potentially significant in relation to the plastic characteristics. Similarly the final temperature and duration of the coking process could possibly exert an appreciable influence upon coke character, the effects being at least partly conditioned by the nature and constitution of the coal charge.

As most of the variables concerned could be mutually disturbing, full investigation of their individual and combined effects was beyond the scope of this project. A realistic and simple compromise was adopted which permitted reasonable appraisal of the potentially important factors. Three independent lines of study were developed in which the character of the resultant coke was assessed and related to (1) charging temperature, (2) final coking temperature and (3) final coking period. Each

TABLE 6.—No. 6 COAL: INFLUENCE OF CHARGING TEMPERATURE ON COKE
(Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period 2 hours)

Oven temp. on charging		Macro-test data of coke									Micro-test data of coke	
		Shatter: % + 1″			Tumbler						Mechanical strength test	
					Stability: % + 1″			Hardness: % + ¼″				
°C±	°F	Initial	Inter.	Final	Initial	Inter.	Final	Initial	Inter.	Final	% + 65m	Ratio $\frac{28\text{-m}}{65\text{-m}}$
25	70	96	77	74	96	73	63	96	89	85	41.1	0.037
205	400	97	88	87	90	73	51	97	88	82	39.9	0.041
260	500	95	84	81	97	88	54	97	92	84	41.7	0.036
430	800	99	87	82	93	75	65	98	89	83	48.5	0.059
540	1000	98	84	70	99	67	43	99	91	86	42.2	0.028
625	1150	88	49	40	85	40	28	98	90	85	—	—
710	1300	88	35	26	95	49	22	99	92	87	48.3	0.067
765	1400	78	24	20	86	34	11	99	92	87	41.3	0.031
790	1450	93	31	26	86	51	39	99	91	85	49.7	0.071
870	1600	67	17	11	82	13	9	99	92	89	55.3	0.134

series of tests was made upon reference pillar samples of standard size consist; from the results of each investigation series appropriate optimum standard conditions were adduced and used in all subsequent studies upon samples from the seam.

Effect of Charging Temperature.— Retorts containing charges of the reference pillar sample were introduced into the oven heated initially to various temperatures ranging from 25°C to 870°C. Thereafter the standard rate of temperature increase was maintained until a final coking temperature of 1010°C was attained and this was held over a period of two hours. The provisional "standards" of 1010°C and two hours for the final phase of coking in this initial test sequence were accepted after due consideration of published data concerning pilot-scale and industrial coking of the Illinois No. 6 seam. Their validity was checked in the immediately succeeding test series.

The details of the study results are summarized in table 6 and figures 24, 25, and 26. It is immediately evident that, as related to shatter index and tumbler stability, the charging temperature assumes critical significance when in the vicinity of 430°C (fig. 24). Coals charged below this temperature produced cokes which exhibited erratic but not drastic variation in these two "quality indices" whereas those charged at successively higher temperatures exhibited a rapid and progressive decline. The rapid decline in shatter and tumbler stability is associated with the closer and more extensive jointing and fine fracturing developed in the cokes formed from coals charged at the higher temperatures. This is made yet more evident in their greatly increased initial and progressive degradation revealed by examination of the initial, intermediate, and final indices for the shatter and tumbler tests (figs. 25 and 26).

It is to be noted that the apparently critical temperature of charging falls within the plastic range. Coals charged below this temperature are heated through the plastic range at a more or less uniform rate, substantially that of the standard rate of temperature increase for the oven (3.6°C/min.). Coals charged at successively higher temperatures above the plastic zone are in effect subjected to an ever-increasing rate of temperature increase from room temperature up to, through, and above the plastic range. Within the plastic range they are afforded less and less time for volume adjustments before the "setting" temperature is reached with an apparent consequent accumulation of stress and the development of increased jointing and fracturing, as indicated by a decrease in macro-mechanical strength.

The tumbler hardness or resistance to abrasion, as well as the micro-mechanical strength indices, can be closely related to the toughness or hardness of the coke substance. This toughness or hardness is consequently revealed as increasing slowly with the rise of charging temperature; the trend is much more strongly marked in the higher ranges (fig. 24).

The contrast in physical appearance and range of macroscopic variation exhibited by the coke series produced in this test is similar to that observed in the No. 5 coal test series as shown in plate 4. Those cokes derived from coals charged at the lower temperatures are relatively dark, of impaired luster, and tough; with increase in charging temperatures the cokes gradually assume a typically metallic gray, lustrous appear-

TABLE 7.—No. 6 COAL: PROXIMATE ANALYSIS OF COKE SAMPLES PRODUCED BY VARYING CHARGING TEMPERATURE
(Reference pillar, standard size consist, rate of temperature increase, final coking temperature and final coking period)

Charging temperature of oven		Air dried							Moisture and ash free			
°C±	°F	Coke yield %*	Moist.	Ash	Vol.	F.C.	S	Btu/lb.	Vol.	F.C.	S	Btu/lb.
25	70	69	2.2	15.9	1.8	80.1	1.17	11746	2.2	97.8	1.43	14342
205	400	62	1.2	14.3	1.1	83.4	1.16	12141	1.3	98.7	1.37	14368
260	500	63	1.4	15.4	1.5	81.7	1.15	11901	1.8	98.2	1.38	14304
430	800	62	1.2	13.9	1.0	83.9	1.17	12247	1.2	98.8	1.38	14425
540	1000	64	1.0	15.0	1.3	82.7	1.23	12030	1.5	98.5	1.46	14321
625	1150	67	0.7	14.5	1.0	83.8	1.14	12169	1.2	98.8	1.34	14350
710	1300	64	0.8	15.6	1.2	82.4	1.18	12007	1.4	98.6	1.41	14362
765	1400	62	0.8	13.1	1.0	85.1	1.17	12392	1.2	98.8	1.36	14393
790	1450	61	0.7	15.1	1.1	83.1	1.23	12061	1.3	98.7	1.46	14324
870	1600	66	0.6	16.0	1.0	82.4	1.23	11952	1.2	98.8	1.47	14331

*Average of three test samples.

ance, but become more brittle and more fragmentary. These differences in physical appearance are not accompanied by any significant variation in chemical properties of the cokes, other than a diminution in the moisture of those formed at higher temperatures (table 7).

The test data demonstrated that both macro-mechanical qualities of the resultant cokes are influenced by the temperature at which the coal is charged. Unfortunately, the optimum qualities in each category are not achieved under the same conditions and in consequence a compromise has to be accepted. For the present series of coking studies, the compromise charging temperature accepted as standard for all samples of the No. 6 coal was 450°C. The significance of the plastic characteristics in relation to the properties of cokes produced from different coal types and constituents under more widely varied but closely controlled conditions of heating, would probably repay exhaustive study.

Effect of Coking Temperature.—To determine the possible influence of final coking temperatures upon coke characteristics, standard charges of reference pillar, medium clarain, and coarse clarain samples were charged at 450°C and by standard rate of temperature increase (3.6°C/min.) brought to final coking temperatures of 930°C, 1010°C and 1090°C in different runs; the final coking period was retained as two hours.

In certain respects the shatter and tumbler test results proved to be rather erratic (table 8 and figs. 27, 28, and 29). Of the cokes formed from the reference pillar samples, the shatter index for that coke having a final temperature of 1010°C proved to be slightly better than that of either of the other two; on the basis of the tumbler indices those cokes formed at 930°C and 1090°C were of greater strength (fig. 27). Cokes produced from the medium clarain at a final temperature of 1010°C proved superior to those of 930°C and 1090°C in all macro-mechanical tests (fig. 28). The coarse clarain at 1010°C final coking temperature produced cokes of superior tumbler stability and slightly reduced tumbler hardness, but the shatter index declined with increase of temperature above 930°C (fig. 29). For each sample group the micro-mechanical strength indices increased slightly with the temperature of final coking, particularly over the range between 930°C and 1010°C.

These test results indicated that for this series of studies a final coking temperature of 1010°C would afford the most generally acceptable "standard."

Effect of Final Coking Period.—The "soaking" or final coking period in this series ranged from one-half hour to ten hours at a "standard" temperature of 1010°C; initial charging temperature was at 450°C ("standard"). The coke test results depict a progressive and corresponding variation in both macro- and micro-mechanical strength with time (table 9, fig. 30). In cokes that had "soaked" from one-half hour to two hours, strength declined, then generally increased with the exception of one shatter and one micro-strength index. The coke which was given the shortest "soaking" time was dark and rather dull; with two hours or more the appearance improved markedly.

It was unfortunate that the minimum strengths determined by the various methods occurred in those cokes "soaked" for two hours, already arbitrarily accepted as "standard" time. However, as it was not practicable to extend each coking run to 7 or 10 hours final coking time so as to achieve the optimum strength, and although the ap-

TABLE 8.—No. 6 COAL: INFLUENCE OF FINAL COKING TEMPERATURE ON COKE FROM SELECTED COAL TYPES
(Standard size consist, charging temperature, rate of temperature increase, and final coking period)

Coal macro-type and coking temp. °C±	°F	Macro-test data of coke: Shatter: % + 1″	Macro-test data of coke: Tumbler: Stability: % + 1″	Macro-test data of coke: Tumbler: Hardness: % + ¼″	Micro-test data of coke: Mechanical strength test: % + 65m	Micro-test data of coke: Mechanical strength test: Ratio 28/65m
Reference pillar						
Coking temp. 930°	1700°	80.8	73.4	87.7	32.3	0.018
Coking temp. 1010°	1850°	84.9	49.3	84.4	40.5	0.027
(Mean standard)		79.8	54.4	85.9		
Coking temp. 1090°	2000°	80.9	73.7	88.3	41.0	0.040
Medium clarain						
Coking temp. 930°	1700°	65.0	53.4	82.3	38.0	0.028
Coking temp. 1010°	1850°	80.3	81.2	88.2	41.4	0.030
Coking temp. 1090°	2000°	79.8	32.2	84.8	43.2	0.044
Coarse clarain						
Coking temp. 930°	1700°	87.0	50.0	86.3	37.4	0.016
Coking temp. 1010°	1850°	83.7	72.6	85.8	42.2	0.017
Coking temp. 1090°	2000°	74.3	52.2	87.2	42.8	0.024

TABLE 9.—No. 6 COAL: INFLUENCE OF PERIOD OF FINAL COKING ON COKE
(Reference pillar, standard size consist, charging temperature, rate of temperature increase, and final coking temperature)

Period of final coking at 1010°C (1850°F) (hours)	Macro-test data of coke: Shatter: % + 1″	Macro-test data of coke: Tumbler: Stability: % + 1″	Macro-test data of coke: Tumbler: Hardness: % + ¼″	Micro-test data of coke: Mechanical strength test: % + 65m	Micro-test data of coke: Mechanical strength test: Ratio 28/65m
½	84.9	68.7	87.5	40.0	0.030
1	88.1	58.6	86.3	39.2	0.024
2	77.3	59.5	87.5	36.4	0.022
4	85.8	70.1	87.6	36.5	0.031
7	82.4	73.0	87.1	38.2	0.045
10	90.9	73.0	87.1	39.0	0.036

pearance of those produced at the shorter coking periods was distinctly unattractive, it was considered desirable to retain the two hour final coking period as "standard" so that all previous test values could be correlated with future results. Although this choice was made it was realized that the coke did not acquire the better qualities that might have resulted from a longer "soaking" period.

On the basis of these three initial study series the coking procedures adopted as "standard" for these investigations concerned with the coking of all pillar, coal type, and seam section samples or fractions thereof for the No. 6 coal were defined as follows:

Charging temperature	450°C
Rate of temperature increase	3.6°C/minute
Final coking temperature	1010°C
Final coking period	2 hours

Influence of Petrographic Constitution of the Macro-types and Seam Sections

In petrographic and chemical constitution, the coal macro-type samples extracted from the original reference pillar do not vary greatly, yet the five types of "standard" samples coked under "standard" conditions yielded cokes which in a number of ways are significantly different (table 10, fig. 31). Of greatest magnitude are the variations in tumbler stability, which rises to a maximum in coke prepared from medium clarain and falls lowest in cokes prepared from the reference pillar and high-vitrain clarain ("vitrain"). Although of less magnitude, the variations of the tumbler hardness index exhibit similar trends.

Starting from the reference pillar cokes, and proceeding through those made from fine, medium, and coarse clarain to those made from "vitrain," the shatter index decreases progressively but erratically (fig. 31). Through the same series, the micro-mechanical strength 65 index increases gradually to a subordinate maximum in the coarse clarain coke, but the micro-strength 28/65 decreases gradually with an anomalous maximum in the medium clarain coke.

Possible relationships between coke characteristics and petrographic composition of the original coal samples were examined (fig. 31). The micro-mechanical strength and tumbler hardness indices appear to fluctuate in harmony with variations in both inertinite proportions and those of mineral matter (which is also non-coking and may be present in a sufficiently fine form and dissemination as to be incorporated with the coke substance). The weakest coke is produced from the high-vitrain clarain or "vitrain" (which is characterized by maximum proportions of vitrinite, minimum proportions of exinite, and greatly reduced content of inertinite and mineral matter). The lower proportion of exinite in the progressively coarser coal macro-types suggests that both of these features may be broadly related to the erratic but general trend toward lower shatter indices exhibited by the coke series shown.

Variation in the petrographic constitution of the seam section samples is of some magnitude. When compared with the mechanical properties of the cokes produced from them, a number of apparent relationships emerge which are significant (tables 3A, 10; fig. 32).

The cokes of greatest shatter and micro-mechanical strength have been derived from the middle seam section samples (II and III) in which the vitri-

TABLE 10.—No. 6 COAL: INFLUENCE OF COAL TYPES AND SEAM SECTIONS ON COKE
(Standard size consist and standard coking conditions)

Coal macrotype or seam section	Macro-test data of coke			Micro-test data of coke	
	Shatter: % + 1″	Tumbler		Mechanical strength test	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Reference pillar (R.P.)	84.9	49.3	84.4	40.5	0.027
(Mean standard)	79.8	54.4	85.9	—	—
Fine clarain (F. C.)	88.0	62.9	85.5	41.5	0.021
Medium clarain (M. C.)	80.3	81.2	88.2	41.4	0.030
Coarse clarain (C. C.)	83.7	72.6	85.8	42.2	0.017
"Vitrain" ("V")	75.3	52.2	83.6	40.6	0.013
Seam section I	76.7	51.6	87.6	41.5	0.022
Seam section II	79.1	60.8	89.1	42.9	0.040
Seam section III	82.4	52.2	85.8	42.4	0.028
Seam section IV	71.7	65.0	85.3	36.7	0.011

TABLE 11.—No. 6 COAL: INFLUENCE OF INDIVIDUAL AND CUMULATIVE SIZE FRACTIONS ON COKE
(Medium clarain, standard coking conditions)

Coal size fraction	Macro-test data of coke			Micro-test data of coke	
	Shatter: % + 1″	Tumbler		Mechanical strength test	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
+6 mesh	61.6	56.1	82.3	44.4	0.027
6 x 10	72.8	68.3	87.1	42.6	0.027
10 x 20	76.3	65.9	86.4	43.3	0.045
20 x 35	89.3	66.6	85.7	41.4	0.025
35 x 65	85.4	81.7	87.7	34.4	0.008
65 x 150	77.4	44.3	80.8	17.5	0.001
−150	17.7	11.1	35.7	0	—
Cumulative size fractions					
All + 35	78.0	46.1	82.6	44.1	0.044
All + 65	73.4	58.9	82.9	43.1	0.038
All + 150	79.7	61.5	83.7	44.8	0.043
Entire sample	80.3	81.2	88.2	41.4	0.030

nite content is lower whereas the exinite and inertinite proportions are higher than in I and IV. Indeed, the degree of relationship (sympathetic and antithetic) exhibited by the variation curves of vitrinite, exinite, and inertinite with those of the micro-mechanical strength and shatter indices is considerable. Further, when the combined proportions of inertinite and mineral matter (which may also be considered as a relatively inert substance insofar as coking is concerned) are graphed, the relationship with the micro-mechanical strength factors is even more closely established. The potential importance of the mineral matter in this connection must clearly depend upon the proportions present, its character, particle size, and distribution; excessive amounts and sizes would certainly be detrimental.

The tumbler hardness index varies progressively in the seam samples but shows a lesser degree of relationship with the petrographic constitution. The tumbler stability values are too erratic for general correlation with maceral distribution.

The over-all coking properties of each of the coal macro-type and seam section samples represents the synthesis of at least three important groups of macerals, present in varying proportions, occurring in different distributions and thus introducing variations in coking characteristics. The results of these preliminary studies have indicated that the coking characteristics of the macerals may be mutually influenced by their individual properties, relative proportions, and size distribution.

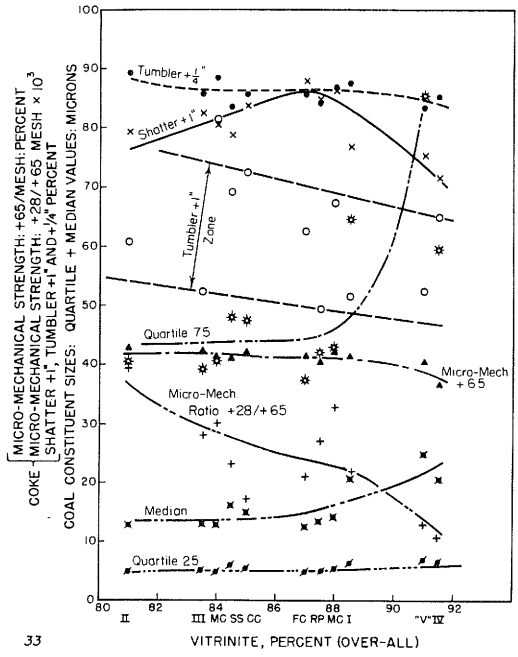
The degree of correlation between petrographic constitution and over-all coking properties of each sample, made evident in the results of macro-type and seam section coking studies, are highly significant.

The macerals vitrinite and exinite are the coking entities of the sample which must be responsible for the "bonding" to form coke during the thermal dissociation. Without these constituents the coal would be largely non-coking, but in the test results there is clear indication that if vitrinite is present in unusually high proportions, weak cokes are produced. Exinite, although present in minor degree, may contribute significantly to the coke properties.

The group maceral inertinite, including a large proportion of disintegrated fusain in the samples used, is generally regarded as non-coking. In these test results there is a strong suggestion that inertinite contributes appreciably to the strength of the coke substance, possibly acting as "aggregate" to the vitrain-exinite "cement." Consequently, it is probable that there are maceral proportion, size, and distribution characteristics which may be associated in an oven charge of suitable particle size consist to yield cokes of optimum strength as determined by the various macro- and micro-mechanical methods.

To investigate this possibility further, graphs have been prepared in which the over-all proportions and constituent sizes of vitrinite and inertinite have been related to coking characteristics for each of the macro-type and seam section samples (tables 3A, 10; figs. 33, 34).

In the vitrinite relation series (fig. 33) the vitrinite quartile 25 exhibits a slight increase in constituent width with greater proportions of that maceral; in samples with more than 87 percent vitrinite, the vitrinite median dimensions increase more definitely, whereas those of the vitrinite quartile



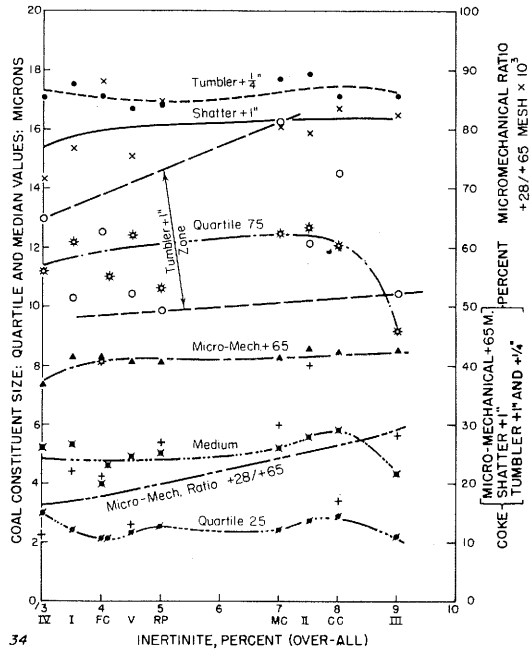


FIG. 33.—No. 6 coal: Vitrinite: proportions and size relations in broken coal types and seam sections, and their influence upon coking characteristics, standard coking conditions (tables 3A and 10).

FIG. 34.—No. 6 coal: Inertinite: proportions and size relations in broken coal types and seam sections, and their influence upon coking characteristics, standard coking conditions (tables 3A and 10).

75 are larger. Those samples with proportions of vitrinite of more than 87 percent owe this characteristic largely to a marked increase in the coarser fragments (tables 2, 3A).

The concurrent variations in the characters of the cokes produced from these samples tend to be erratic but are significant. As regards resistance to shatter, the optimum coke was produced from samples with 87 percent vitrinite, the critical value beyond which the constituent size increased markedly. Both tumbler hardness and micro-strength 65 index decreased slowly with increase of vitrinite, a trend slightly more evident in the range above 90 percent of this maceral. The micro-strength 28/65 value decreased more precipitately with increase of vitrinite, a trend again more evident in the higher-vitrinite samples. Tumbler stability appeared to vary in an erratic manner, generally decreasing with increased vitrinite.

The inertinite relation series (fig. 34) reveal different features. Included with the inertinite is the fusain content of each sample. Mechanical disintegration studies of this maceral from the No. 6 seam revealed that in the course of sample preparation, approximately 70 percent of the material broke down to pass through a 150-mesh sieve; microscopic examination confirmed that much of the minus 150-mesh fraction was of much smaller dimensions. Consequently, it is not unexpected that the quartile 25, median, and quartile 75 dimensions, after a slow and uneven rate of increase with increasing inertinite proportions,

decline quite sharply in the vicinity of 8 percent inertinite total content. It is probable that the increased proportions of this constituent are derived from originally coarse lenticles of fusain that disintegrated under the conditions of sample preparation. As the proportions of inertinite increase from 3 to 9 percent in the sample, the shatter index, tumbler hardness, and micro-mechanical strength, although erratic, demonstrate a slow but general improvement. The tumbler stability trend is still less well defined but tends to increase with inertinite content.

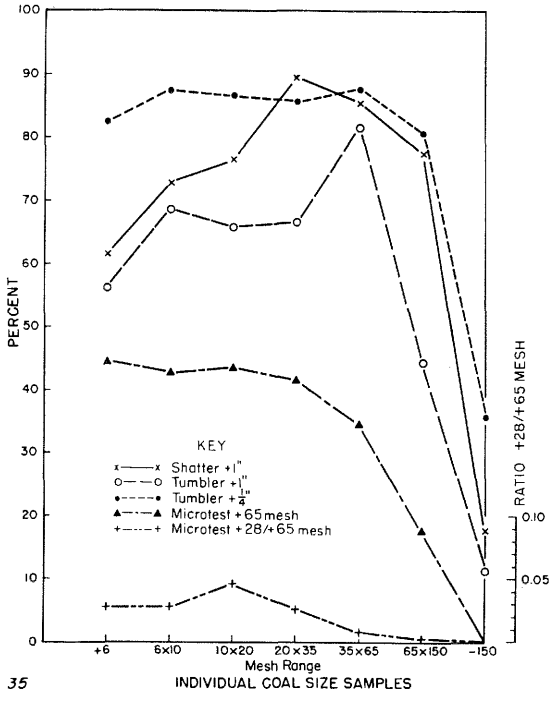
From the results of the petrographic study of the nine macro-type and seam samples in relation to the mechanical properties of the coke produced from them, it appears that the maceral proportions, their size characteristics, and distribution in the broken coal may be of considerable importance in conditioning coke quality.

Influence of Coal Particle Size Fractions

Petrographic analyses of individual size fractions of all macro-type and seam samples demonstrated that coal breakage during preparation induced a measure of selective variation in the proportions and sizes of the macerals present in the different size fractions of each standard sample (table 2). The extent of variation appeared to depend largely upon the original character of the sample, that is, the nature, proportions, and sizes of the original macerals. Consequently, although highly desirable, it was impossible to secure complete uniformity of distribution of the coal constituents in the various size fractions of a single sample. The results of the present study series must be considered as influenced by both coal particle size characteristics in the oven charge and some degree of variation in petrographic constitution of the size fractions.

The initial study was concerned with the coking characteristics of each particle size fraction produced from a single sample of medium clarain (table 11, figs. 35, 36, 37, and pl. 2). The relations between coke characteristics and each particle size fraction of the coal are well defined (fig. 35). From the coke produced from the coarsest fraction of coal (plus 6-mesh), the shatter index improves remarkably to a maximum for the coke from the 20 x 35 mesh size, declines at a comparable rate to the product of the 65 x 150 mesh coal, and decreases very greatly to that representing the minus 150-mesh fraction. After an initial improvement in the coke of the 6 x 10 mesh coal, tumbler hardness and stability are both slightly reduced in the cokes of the 10 x 20 and 20 x 35 mesh fractions, increased to maximum values for the product of the 35 x 65 mesh fraction, and then decline rapidly to the coke of the minus 150-mesh fraction. With one exception in each case, the micro-mechanical coke strength indices decrease slowly with mesh size of the coal as far as the 20 x 35 mesh fraction; the smaller mesh fractions exhibit a rapid decline to zero for the cokes formed from minus 150-mesh material.

In order to assess the influence of petrographic difference among the size fractions on the drastic differences in coke quality, the petrographic proportions of the four main seam constituents have been recorded in figure 36, together with the coke characteristics. With the possible exception of the 10 x 20 mesh fraction (in which the proportions of vitrinite, inertinite, and mineral matter departed rather severely from the normal trend), the petrographic variation is more or less uni-



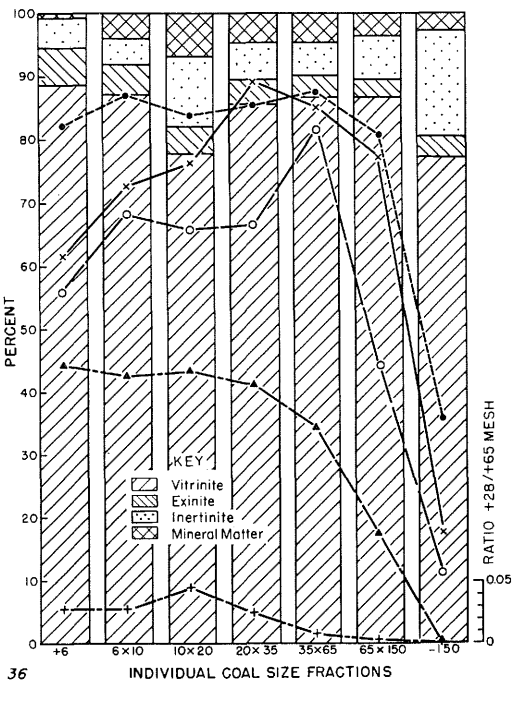


FIG. 35.—No. 6 coal: Character of coke produced from individual size fractions. Medium clarain, standard coking conditions (table 11).

FIG. 36.—No. 6 coal: Character of coke produced from individual size fractions related to petrographic constitution (vitrinite, exinite, inertinite, and mineral matter). Medium clarain, standard coking conditions (tables 3B, 11).

formly progressive up to the 65 x 150 mesh fraction; for the minus 150-mesh fraction the "non-coking" maceral inertinite increased greatly (by 10 percent approximately), whereas the coking constituents vitrinite and exinite decreased correspondingly. With the exceptions already noted, all other fractions are constituted of maceral proportions similar to those which yielded satisfactory cokes in the previous test series.

It thus appears that the considerable variation in mechanical properties exhibited by the cokes of the individual coal size fractions of the medium clarain (fig. 36) cannot be attributed significantly to petrographic differences. The possible effect of the combined influence of two or more macerals has not been explored, hence although the variation in coke characteristics appear to be substantially a function of coal particle size in this series of tests, the possibility of the presence of other factors should not be ignored.

Although particle sizes vary within each fraction, such variation is restricted as compared with that of the entire standard sample. In fragment aggregates of uniform particle size, the smaller the particle size the greater is the available pore space, because of the higher ratio of surface area to volume and mass. This ratio conditions such factors as adhesion, friction, and "bridging." It is probable that the more rapid development of these effects in the minus 65-mesh size fractions is a factor in the decline of coke quality in this

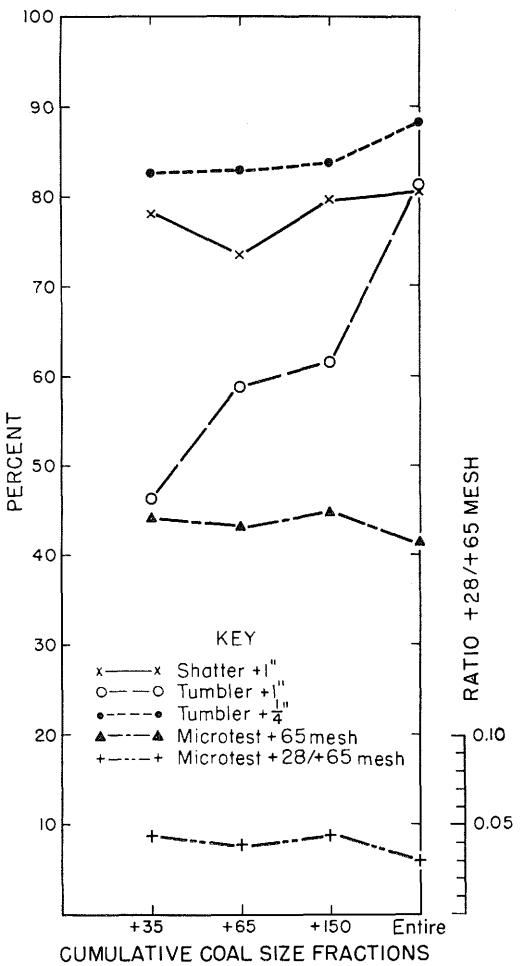


FIG. 37.—No. 6 coal: Character of coke produced from cumulative size fractions. Medium clarain, standard coking conditions table 11).

range. The decline of macro-mechanical strength (shatter index and tumbler stability) in cokes derived from the coarser coal fractions may be related to the limitation of contact areas along which fusion and "particle-welding" can take place during coking. In both cases the *micro*-mechanical strength factors showed no corresponding decline, the nature and scale of the micro-strength tests being primarily concerned with the coke produced from the substance of the individual particles.

The importance of the finer fractions as space fillers and bonding elements in the coke oven charge was made evident by studies involving the coking of cumulative size fractions from standard charges (table 11, fig. 37). As each of the successive finer fractions was added to the coal charge, the quality of the coke produced was generally improved, most notably in terms of the tumbler stability and, with one exception, in each of the shatter and micro-mechanical strength indices.

Influence of Coal Size Consist

The differing mechanical properties exhibited by cokes formed from individual and cumulative size fractions of a single standard sample gave added interest to the investigation of the importance of the over-all coal size consist.

The successful development and industrial application of methods of control of coal size consist of the oven charges with accompanying important modifications of petrographic distribution has been reported by Burstlein (1955). The results achieved with coals of the Saar, Lorraine, and the Ruhr using a size consist preferentially weighted in the coarser and finer fractions (low proportions of middle sizes) have been extremely good. As, however, the coking characteristics of coal beds are affected by both petrographic and rank variation, it is probable that optimum conditions of size consist will vary with different coals.

For the purposes of this study series, standard samples of the reference pillar were subjected to secondary preparation by selective crushing and screening, so as to vary the size consist (i.e. proportions of the different sizes) within the standard size range (table 12).

TABLE 12.—No. 6 COAL: SIZE DISTRIBUTION, INDIVIDUAL AND CUMULATIVE, RESULTING FROM SELECTIVE AND STANDARD BREAKAGE
(Proportions in percentage)

Sample*	(a)		(b)		(c)		(d)†		(e)		(f)		(g)		(h)		(i)	
Ratio $\frac{+65}{-65}$	0.6		2.2		4.0		15.7		1.1		3.7		4.6		6.0		7.6	
Size range mesh	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+6	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—
6 x 10	4.33	4.33	17.3	17.3	46.43	46.43	49.63	49.63	6.7	6.7	2.8	2.8	26.37	26.37	13.25	13.25	27.54	27.54
10 x 20	6.15	10.48	17.2	34.5	7.21	53.64	25.32	74.95	2.9	9.6	23.3	26.1	13.68	40.05	32.89	46.14	30.75	58.29
20 x 35	4.47	14.95	17.3	51.8	2.14	55.78	12.34	87.29	8.0	17.6	30.7	56.8	20.93	60.98	26.81	72.95	19.82	78.11
35 x 65	22.68	37.63	17.2	69.0	23.80	79.58	6.33	93.62	34.8	52.4	21.9	78.7	20.86	81.84	12.85	85.80	10.33	88.44
65 x 150	36.27	73.90	17.2	86.2	11.58	91.16	3.68	97.30	26.5	78.9	11.3	90.0	10.40	92.24	6.95	92.75	5.68	94.12
−150	26.10	100.00	13.8	100.0	8.84	100.00	2.70	100.00	21.1	100.0	10.0	100.0	7.76	100.00	7.25	100.00	5.88	100.00

TABLE 12.—(Concluded)

Sample*	(j)		(k)		(l)		(m)		(n)‡		(o)		(p)		(q)	
Ratio $\frac{+65}{-65}$	10.2		11.9		3.9		10.3		11.8		4.8		10.5		11.3	
Size range mesh	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+6	—	—	—	—	2.01	2.01	1.43	1.43	23.27	23.27	—	—	—	—	0.15	0.15
6 x 10	40.05	40.05	50.79	50.79	5.10	7.11	34.73	36.16	27.27	49.54	1.62	1.62	17.46	17.46	47.72	47.87
10 x 20	26.99	67.04	23.60	74.39	26.93	34.04	29.74	65.90	20.98	70.52	27.67	29.29	47.32	64.78	25.10	72.97
20 x 35	16.07	83.11	11.51	85.90	28.01	62.05	16.55	82.45	13.86	84.38	36.97	66.26	18.38	83.16	12.31	85.28
35 x 65	7.90	91.01	6.35	92.25	17.58	79.63	8.81	91.26	7.86	92.24	16.61	82.87	8.17	91.33	6.60	91.88
65 x 150	4.31	95.32	3.84	96.09	10.17	89.80	4.92	96.18	4.40	96.64	8.15	91.02	4.09	95.42	3.98	95.86
−150	4.68	100.00	3.91	100.00	10.20	100.00	3.82	100.00	3.36	100.00	8.98	100.00	4.58	100.00	4.14	100.00

*Reference pillar—A through N; medium clarain—O through Q.
†Standard sample prepared for laboratory coke.
‡Prepared by standard procedure as normally used for pilot oven charge.

The specific method of preparation for each sample need not be detailed but through progressive screening care was taken to avoid unnecessary crushing or particle shattering. Five laboratory study series were completed (table 13) of which 1, 2, and 4 were most closely related in coking conditions; series 3 and 5 were poorly related and anomalous factors were present which made the correlation of results difficult.

In order to examine systematically the effect of size consist of the coal charge upon the mechanical characteristics of the cokes, it was necessary to have some concise method of representing significant coal size characteristics in the diagrams showing variation of the coke properties. The tests on the effects of coking the individual coal size fractions had demonstrated that for coal below 65-mesh, the coking qualities deteriorated rapidly. Consequently, the ratio of the proportions of plus 65-mesh coal to minus 65-mesh coal was accepted as an appropriate and convenient but not entirely satisfactory method of representing the size consist of each sample.

The first test series dealing with the effects of coal size consist in the oven charge included four reference pillar samples, of which one (d) was used in its original condition (it represented a standard sample). Of the other three, the first (a) was given secondary preparation so as to produce a large proportion of fines; the second (b) was made to yield equal proportions of all size fractions; the third (c) was produced so as to conform with the size distribution used so successfully by Burstlein, in which the larger and smaller sizes are emphasized while the middle size ranges are subordinated (table 12, fig. 38).

The mechanical strength characteristics of the cokes prepared from these four coal charges exhibit interesting and systematic variations (table 13, fig. 39). That charge in which the "fines" (minus 65-mesh) were preponderant (a), gave lowest values for all strength indices except that of tumbler hardness. With increase in the proportion of plus 65-mesh coal fractions, all coke strength indices except the tumbler hardness were improved, especially those of shatter and tumbler stability.

The coal sample prepared with substantial and nearly equal proportions of both coarse and fine fractions and subordinate amounts of the intermediate fractions (c) showed further improvement in the shatter index, slight increase in the tumbler hardness and one micro-mechanical strength factor (ratio 28/65), but a deterioration in the second micro-strength index and the tumbler stability, the latter being quite severe.

In all respects except that of tumbler hardness, the coke produced from the standard sample (d) proved to have the greatest strength.

Series 2, 3, 4, and 5 (table 13) were used to explore further possibilities of controlling coke character through the size consist of the oven charge. The results of series 2 and 4 (table 13, figs. 39, 40), although differing in degree, confirm those of series (1); no definite explanation can be offered for the apparent anomalies, but they may be due to lack of homogeneity as a result of size segregation in the retort charges. A similar explanation may account for the apparently anomalous results obtained from the examination of some of the cokes of test series 3 and 5 (table 13, figs. 41, 42, 43).

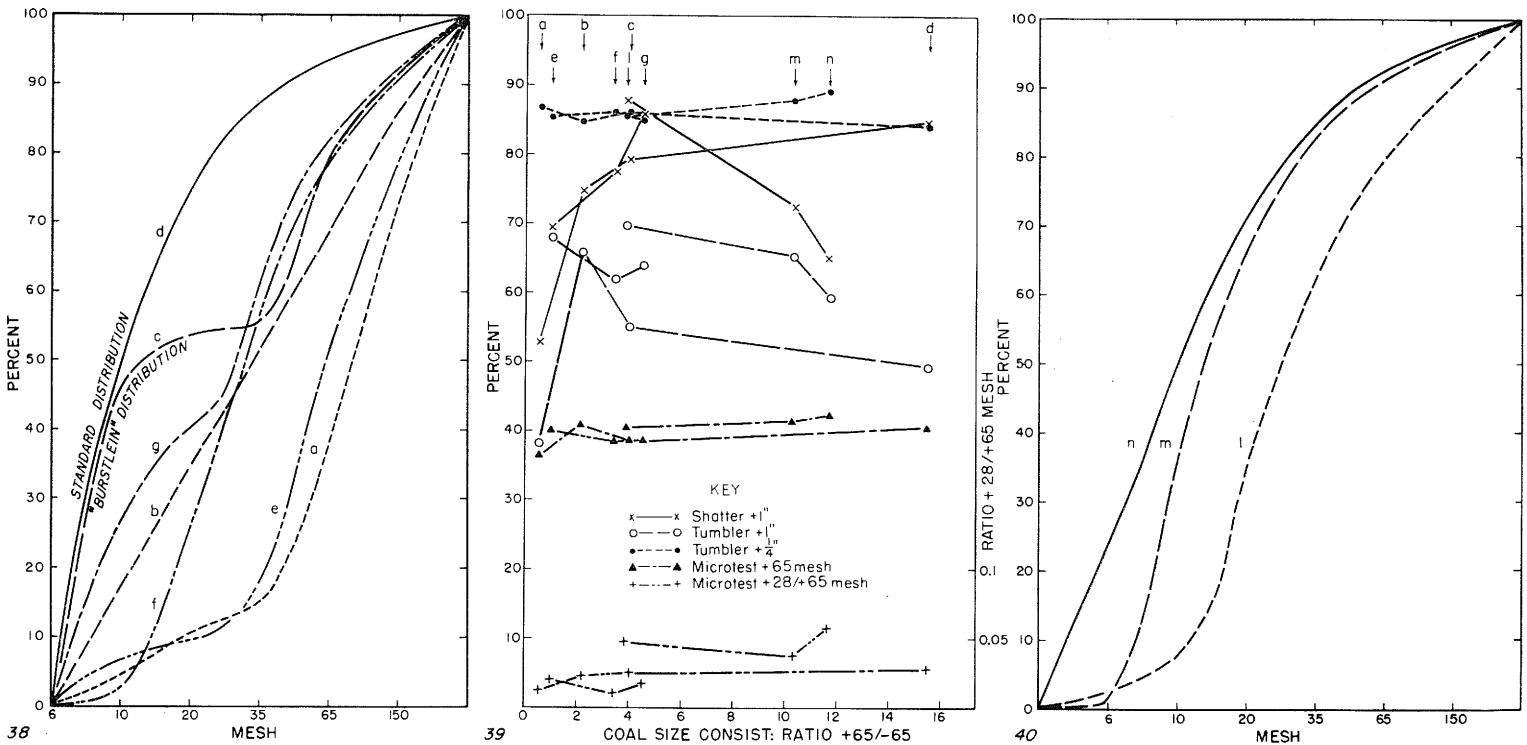


FIG. 38.—No. 6 coal: Influence of selective breakage on distribution of particle size for series 1 and 2. Reference pillar. Use with fig. 39 (table 12).

FIG. 39.—No. 6 coal: Influence of size consist on coke for series 1, 2, and 4 (for series 3 and 5 see figs. 42 and 43). Reference pillar, standard coking condition. Use with figs. 38 and 40 (table 13).

FIG. 40.—No. 6 coal: Influence of selective breakage on distribution of particle size for series 4. Reference pillar. Use with fig. 39 (table 12).

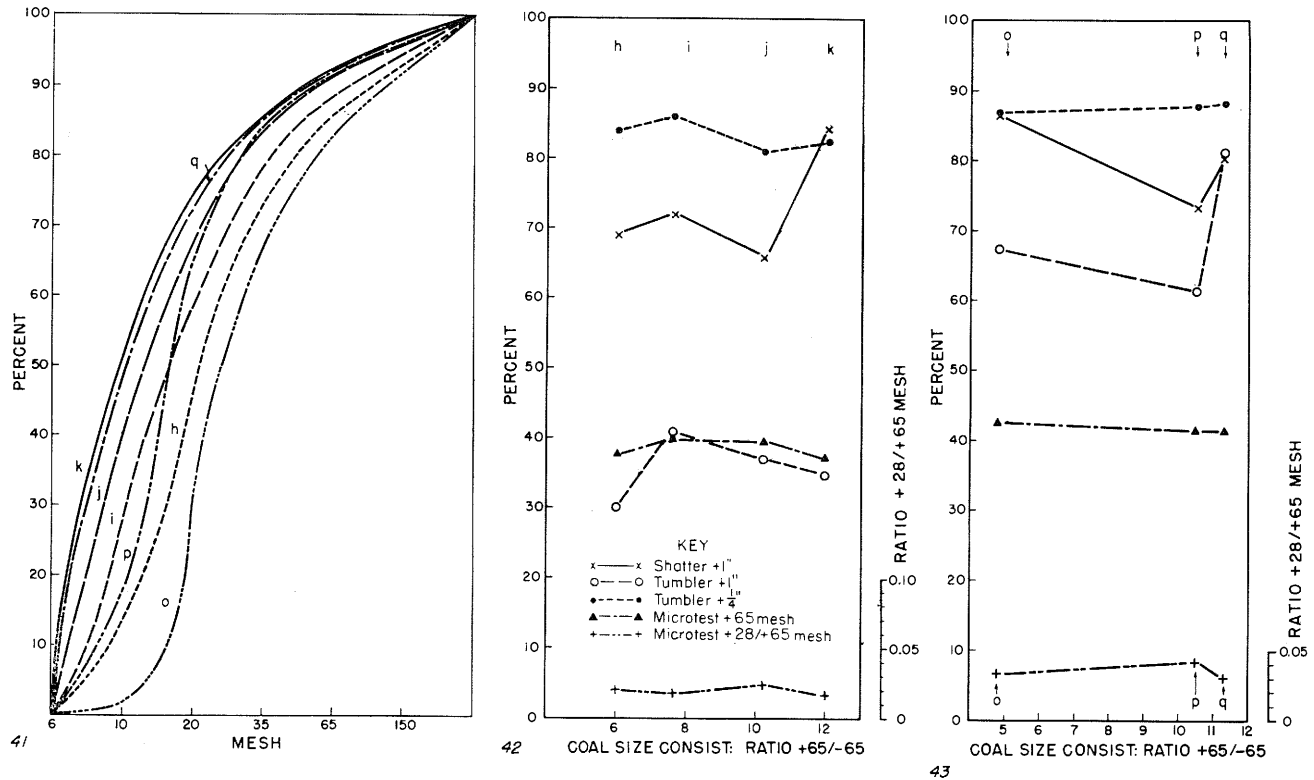


FIG. 41.—No. 6 coal: Influence of selective breakage on distribution of particle size for series 3 and 5. Reference pillar and medium clarain. Use with figs. 42 and 43 (table 12).

FIG. 42.—No. 6 coal: Influence of size consist on coke for series 3. Reference pillar, standard coking conditions. Use with fig. 41 (table 13).

FIG. 43.—No. 6 coal: Influence of size consist on coke for series 5. Medium clarain, standard coking conditions. Use with fig. 41 (table 13).

TABLE 13.—No. 6 COAL: INFLUENCE OF SELECTIVE AND STANDARD BREAKAGE ON COKE
(Standard coking conditions)

Coal size "factor" Ratio $\frac{+65}{-65}$	Macro-test data of coke			Micro-test data of coke	
	Shatter:	Tumbler		Mechanical strength test	
	% + 1″	Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Series 1. Reference pillar					
(a) 0.6	52.9	38.3	86.8	36.6	0.011
(b) 2.2	74.3	65.8	84.8	40.8	0.024
(c) 4.0	79.4	55.0	86.1	38.8	0.026
(d) 15.7	84.9	49.3	84.4	40.5	0.027
Series 2. Reference pillar					
(e) 1.1	69.5	68.0	85.3	40.1	0.020
(f) 3.7	77.5	62.0	86.0	37.4	0.011
(g) 4.6	86.0	64.0	85.0	38.9	0.017
Series 3. Reference pillar					
(h) 6.0	69.0	30.0	84.0	37.7	0.020
(i) 7.6	72.0	41.0	86.0	40.0	0.017
(j) 10.2	66.0	37.0	81.0	39.3	0.023
(k) 11.9	84.5	35.0	83.0	37.5	0.017
Series 4. Reference pillar					
(l) 3.9	87.9	69.6	85.7	41.3	0.047
(m) 10.3	72.5	65.2	87.9	41.2	0.038
(n) 11.8	64.6	59.3	89.4	42.6	0.057
Series 5. Medium clarain					
(o) 4.8	86.3	67.2	86.4	42.5	0.033
(p) 10.5	73.4	61.3	87.8	41.5	0.042
(q) 11.3	80.3	81.2	88.2	41.4	0.030

TABLE 14.—No. 6 COAL: INFLUENCE OF SIZE CONSIST ON PILOT OVEN AND LABORATORY COKE
(Full seam bulk sample, standard coking conditions)

Coke preparation and Coal size "factor"				Macro-test data of coke			Micro-test data of coke	
				Shatter:	Tumbler		Mechanical strength test	
Sample	+ 8/−8	+20/−20	+65/−65	% + 1″	Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Laboratory Coke and Laboratory Test Results								
(l)	0.06	0.5	3.9	87.9	69.6	85.7	41.3	0.047
(m)	0.33	1.9	10.3	72.5	65.2	87.9	41.2	0.038
(n)	0.64	2.4	11.8	64.6	59.3	89.4	42.6	0.057
Pilot Oven Coke and Plant Scale Test Results								
1.	0.06	0.5	3.9	91.2	25.9	67.9		
2.	0.33	1.9	10.3	93.0	24.5	63.9		
3.	0.64	2.4	11.8	86.0	11.8	65.4		
4.	1.23	2.1	—	85.0	14.4	58.7		

From these studies it is apparent that coal size consist (as well as size range) is an important factor in determining coke character. Certain trends have appeared but cannot be considered as universally applicable; it is quite probable that optimum values for different strength indices may be produced by different size consists. For each coal there is apparently an optimum, "compromise" size consist, reasonably simple to obtain economically, which will permit the production of coke of most nearly satisfactory quality for particular purposes.

In consideration of the results of these studies and the relative ease and simplicity of the preparation procedure, the size consist of the samples as originally prepared was accepted as "standard" for this coal in all subsequent runs.

Comparison of Laboratory and Pilot Oven Cokes

Of the various studies developed in the course of this project only one offered possibilities of exploration on pilot-plant scale with existing equipment, namely that relating to the effects of coal size consist upon coke character. The limited breaking and sizing facilities available, however, conditioned the method, degree, and control of preparation.

Both pilot oven and laboratory cokes were produced from three identical representative seam samples of different size consists and assessed by the relative standard procedures (table 14, fig. 44). Unfortunately, the standard screens used for determining size consist in the pilot plant operations did not correspond exactly with those used in the laboratory. Consequently, it was necessary to adjust the coal size consist "measure" to secure a common basis of comparison; instead of the ratio of +65/–65 there was substituted the ratio +20/–20.

In the preliminary discussion of the techniques developed for this project it was considered to be neither practicable nor necessary to establish coking and testing methods which conjointly would yield results numerically identical with those obtained on the industrial-scale operation. It would be sufficient to develop those methods of coking and assessment in the laboratory which would establish trends capable of correlation with those obtained in commercial practice.

These objectives were substantially achieved. Although the results of corresponding tests upon the laboratory and pilot oven coke differ markedly in absolute values, the trends which emerge from the results of each of the comparable methods of examination show an encouraging degree of similarity (fig. 44).

Influence of Fusain

One of the problems of major concern to the coal-producing industries has been the utilization or disposal of the fine fractions remaining after preparation. Generally, these have been reported to be high in finely divided fusain and mineral matter, both of which are essentially non-coking.

The micro-petrographic analyses of the various size fractions produced in the standard preparation of macro-type and seam samples used in this project has demonstrated a marked increase of inertinite in the finer fractions, especially the minus 150-mesh. Examination of the cokes produced from type and seam sections together with the results of the cumulative size fraction coke studies has suggested that, although non-cok-

TABLE 15.—No. 6 FUSAIN: CHEMICAL ANALYSES AND SIZE DISTRIBUTION OF AGGREGATE SAMPLES FROM MASSIVE LENTICLES OF FUSAIN

(Each sample broken between rolls at 3 mm)

Sample, fraction and/or condition	As received				Moisture and ash free								Size distribution	
	Proximate				Proximate		Ultimate						%	Cum. %
	Moist.	Ash	Vol.	F. C.	Vol.	F. C.	H	C	N	O	S	Btu/lb.		
Fusain, sample A. +6 mesh	3.1	11.9	20.4	64.6	24.0	76.0	3.83	84.89	0.76	6.56	3.96	14359	0.69	0.69
6 x 10	3.1	14.8	18.8	63.3	22.9	77.1	3.71	86.15	0.77	3.35	6.02	14490	4.47	5.16
10 x 20	2.9	14.9	17.9	64.3	21.8	78.2	3.52	85.92	0.83	2.51	7.22	14659	7.57	12.73
20 x 35	2.8	16.5	15.5	65.2	19.2	80.8	3.18	85.92	0.68	1.43	8.79	14622	6.86	19.59
35 x 65	2.0	16.3	13.6	68.1	16.6	83.4	2.94	86.85	0.58	0.62	9.01	14777	5.18	24.77
65 x 150	1.7	14.7	11.6	72.0	13.9	86.1	2.74	88.51	0.41	0.14	8.48	14554	5.10	29.87
−150	1.1	4.3	8.4	86.2	8.9	91.1	2.58	93.37	0.31	1.85	1.89	14940	70.13	100.00
Fusain, sample B −150	1.1	8.6	10.6	79.7	11.7	88.3	2.69	91.16	0.41	2.78	2.96	14819		

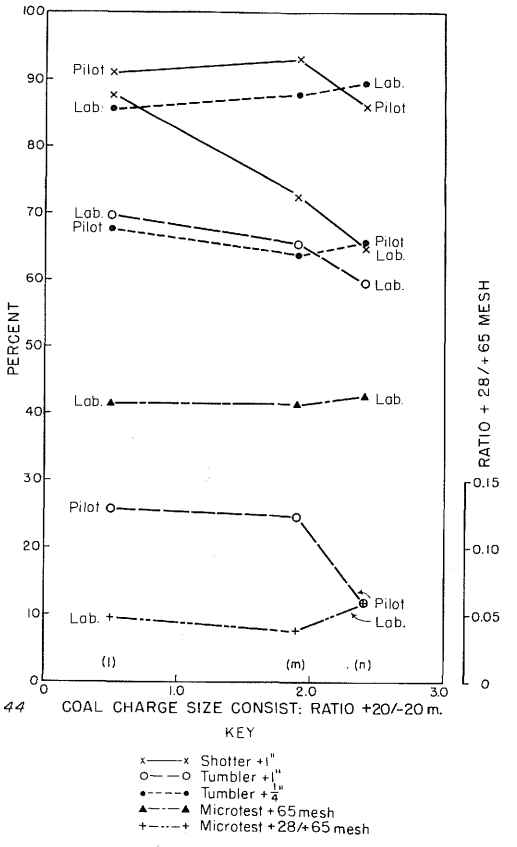


FIG. 44.—No. 6 coal: Comparison of laboratory and pilot oven cokes, produced from the same coal sample and assessed by laboratory and pilot scale techniques respectively. Reference pillar, standard coking conditions (table 14).

ing, the inertinite and related constituents of the coal seam contribute significantly to the characteristics of the cokes produced.

In order to investigate the magnitude of the contribution to coke properties made by inertinite, a series of studies was developed in which additional fusain from the same seam (No. 6 coal) was blended in varying proportions with macro-type samples at standard and reduced size consists.

Fusain Sample Preparation.—The fusain used in this test series was an aggregate sample obtained from lenses, up to two inches thick and several square feet in area occurring as abnormal developments in a well defined and persistent fusain horizon of the No. 6 seam in the same area as that from which the pillar was extracted. Each lens comprised a great number of individual fragments of varied character together with minor proportions of vitrain in thin, erratic sheets. The material was excavated from the seam directly into cans and sealed against moisture loss and oxidation.

Before preparation, the fusain was hand picked to remove extraneous matter including vitrain, and visible mineral matter such as clay partings and pyrite. It was then passed once only between rolls set at 1/16 inch, followed by air drying to remove bed moisture. The size consist of the broken fusain is presented in figure 45 which shows the heavy development of fines. Although crushed at 1/16 inch, more than 70 percent passed the 150-mesh screen. Chemical analyses were made of each size fraction (table 15, fig. 46).

The remarkable range of chemical variation exhibited by this carefully selected fusain sample is in accordance with the results of other examinations (Marshall, 1954); it is surprising, however, that it appears so prominently in size fractions prepared by a mechanical process. The carbon content increases rapidly in the fractions below 20-mesh, whereas moisture, volatile, and hydrogen decrease over the whole range of increasing fineness of division. Below 20-mesh, the nitrogen likewise decreases progressively. Ash and sulfur increase sympathetically to the 35 x 65 mesh range and then decline into the finest sizes. Pyrite is present as finely divided bodies often occupying cell cavities.

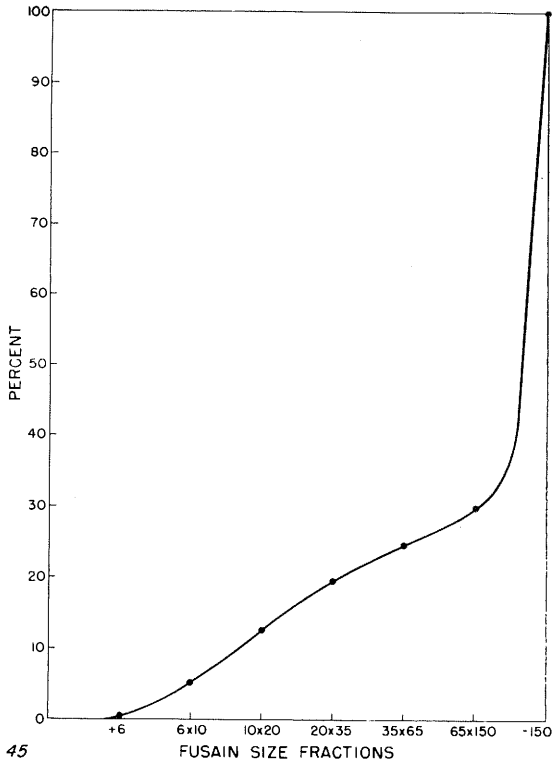
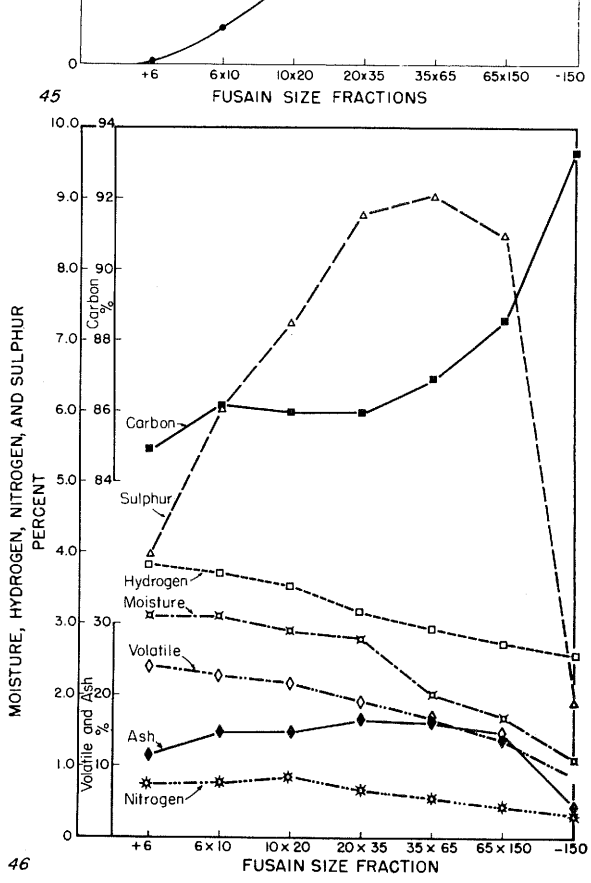


FIG. 45.—No. 6 coal: Size distribution curve of fusain broken at 1/16 inch (1.5 mm.) for blending with seam macrotypes (table 15).

FIG. 46.—No. 6 coal: Variation in chemical composition of size fractions of fusain broken at 1/16 inch (table 15).

FIG. 46A.—Micrometric analysis of No. 6 coal and No. 5 fusain used in blending with various coal samples.



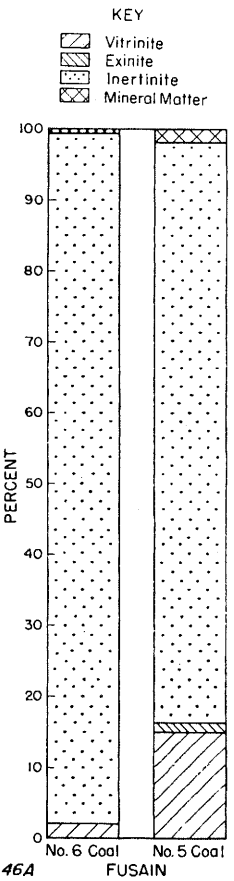


TABLE 16.—No. 6 COAL: SIZE DISTRIBUTION OF MEDIUM CLARAIN PREPARED BY RE-BREAKING STANDARD SIZE CONSISTS
(Samples used for blends with minus 150-mesh fusain of the same coal)
(Proportion in percentage)

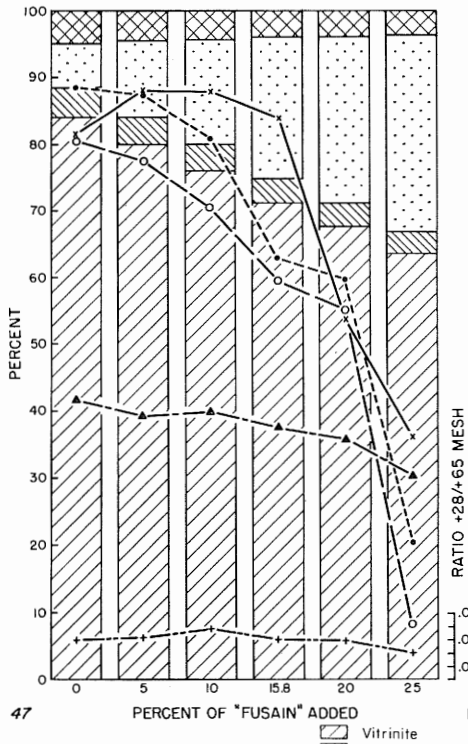
Coal size (mesh)	Medium clarain standard		Medium clarain reduced at 1/16″		Medium clarain reduced at 1/32″	
	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+6	0.15	0.15	0	0	0	0
6 x 10	47.72	47.87	17.46	17.46	1.62	1.62
10 x 20	25.10	72.97	47.32	64.78	27.67	29.29
20 x 35	12.31	85.28	18.38	83.16	36.97	66.26
35 x 65	6.60	91.88	8.17	91.33	16.61	82.87
65 x 150	3.98	95.86	4.09	95.42	8.15	91.02
−150	4.14	100.00	4.58	100.00	8.98	100.00

Of these widely varying fractions only the minus 150-mesh fusain was selected for the purpose of blending with the seam samples. Micro-petrographic analysis of this portion of the fusain is shown in figure 46A (table 3A).

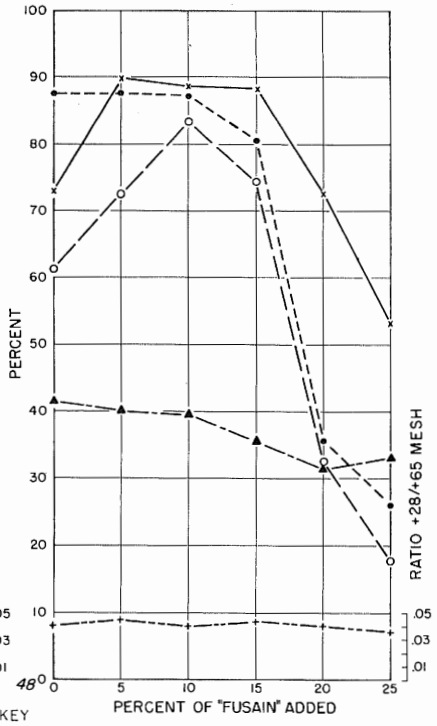
Medium Clarain Sample Preparation. These studies were made in order to examine the nature of the effects of the fusain upon cokes prepared from fine coal, with special concern for the possibility of utilizing "fines" independently for the production of coke. This appears to be of some importance in view of the considerable amounts of such material now produced as a direct result of mechanization and high production. Consequently, in addition to the medium clarains of standard size consist, two series of samples were prepared by re-crushing the standard at 1/16 inch and 1/32 inch roll separation respectively. The size consists of each of these series was considerably reduced as compared with the standard (table 16).

Blends of Medium Clarain and Minus 150-mesh Fusain.—Each blend was prepared immediately before coking, the appropriate proportions of medium clarain and minus 150-mesh fusain being thoroughly mixed and charged with the minimum possible segregation. "Standard" coking procedures were used but special precautions had to be taken after quenching the cokes with the highest fusain contents so as to recover all "unincorporated" fine material. A control sample without blended fusain was included in each series.

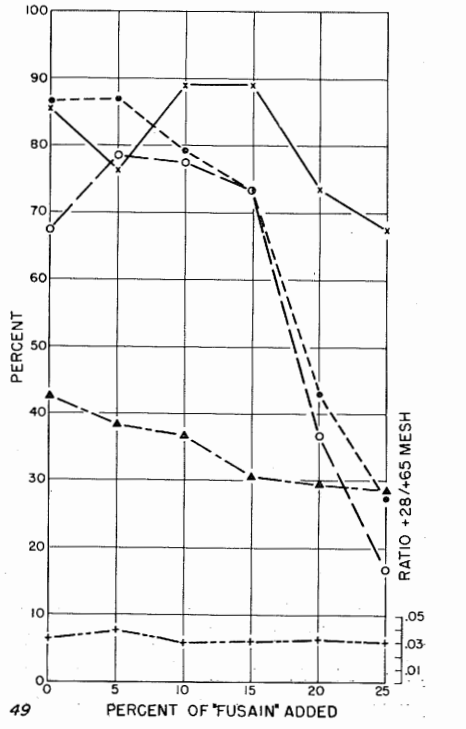
The results of the tests are shown in plate 3, table 17, and figures 47, 48, and 49. When added to standard samples of medium clarain so as to constitute up to 10 percent of the retort charge, fusain produced in the cokes significant improvement in both shatter index and the micro-mechanical strength 28/65 index (fig. 47). With increasing fusain proportions, both of these tests demonstrated a deterioration which, with


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED
Vitrinite
Exinite
Inertinite
Mineral Matter

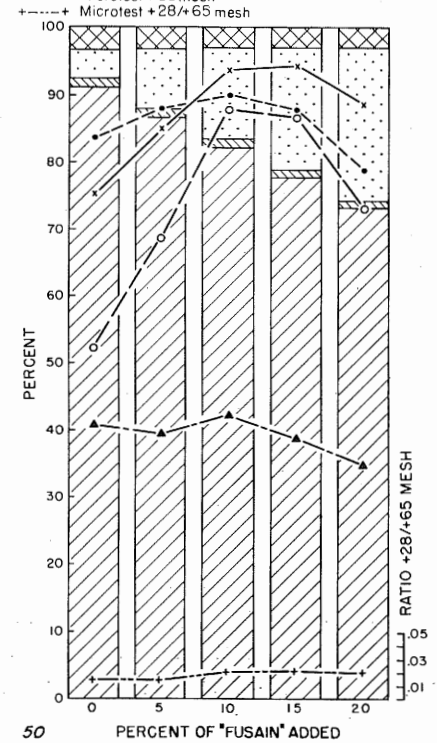
47


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED
KEY
Shatter +1"
Tumbler +1"
Tumbler +¼"
Microtest +65 mesh
Microtest +28/+65 mesh

48


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED

49


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED

50

more than 15 percent additional fusain, became quite catastrophic so far as the shatter index was concerned. Both tumbler stability and tumbler hardness, as well as the micro-strength 65 index, declined with each addition of fusain.

Blends between minus 150-mesh fusain and the clarain samples re-crushed at 1/16 inch (of reduced size consist) produced cokes of rather different nature (fig. 48). Shatter index and tumbler stability were substantially improved with fusain contents of as much as 10 and 15 percent respectively, but fell precipitously with further increases in the proportions of fusain. The micro-strength 28/65 index and tumbler hardness were substantially consistent up to the same respective proportions and thereafter declined, the latter at a rapid rate. The micro-mechanical 65 index decreased with increased fusain content in the coal charge almost throughout the test series, but only slightly up to 10 percent fusain added.

Still further reduction in the size consist of the medium clarain (re-crushed at 1/32 inch) with which the minus 150-mesh fusain was blended, established similar trends (fig. 49). Deterioration in the tumbler stability however appeared in all blends with more than a 5 percent content of additional fusain but not markedly up to 15 percent added. Decline in tumbler hardness began at lower concentrations of additional fusain (above 5 percent) and the decrease in micro-mechanical strength was more accentuated.

Blends of High-vitrain Clarain ("Vitrain") and Minus 150-mesh Fusain.—With high-vitrain clarain ("vitrain") the effects of the additional minus 150-mesh fusain upon quality of coke produced are substantial. Unfortunately shortage of sample material restricted the study to one series with the "vitrain" of standard size consist (table 17).

All methods of mechanical assessment demonstrated remarkable improvements of coke quality with increasing additional fusain content up to 10 percent in the coal charge (fig. 50); the proportionate improvements in shatter, tumbler, and micro-mechanical indices were the greatest recorded. Indeed, some of the cokes proved almost impossible to break under the conditions of test. Additional fusain contents in excess of 15 percent were accompanied by deterioration in all coke strength indices but this was of less consequence than in the corresponding cokes of the medium clarain-fusain blends.

From these results it would appear that generally improved mechanical properties may be expected from normal coal blends with fusain in proportions up to 10 or 15 percent, provided that the normal coal is of a preferred size consist which may be characteristic of the seam. In each series the improvements were such as to produce optimum cokes of substantially the same mechanical properties.

FIG. 47.—No. 6 coal: Influence of fusain on coke. Medium clarain, standard size consist, fusain minus 150-mesh, standard coking conditions (table 17).

FIG. 48.—No. 6 coal: Influence of fusain on coke. Medium clarain, reduced size consist (re-broken at 1/16 inch), fusain minus 150-mesh, standard coking conditions (table 17).

FIG. 49.—No. 6 coal: Influence of fusain on coke. Medium clarain, reduced size consist (re-broken at 1/32 inch, fusain minus 150-mesh, standard coking conditions (table 17).

FIG. 50.—No. 6 coal: Influence of fusain on laboratory coke. "Vitrain" (high-vitrain clarain), standard size consist, fusain minus 150-mesh, standard coking conditions (table 17).

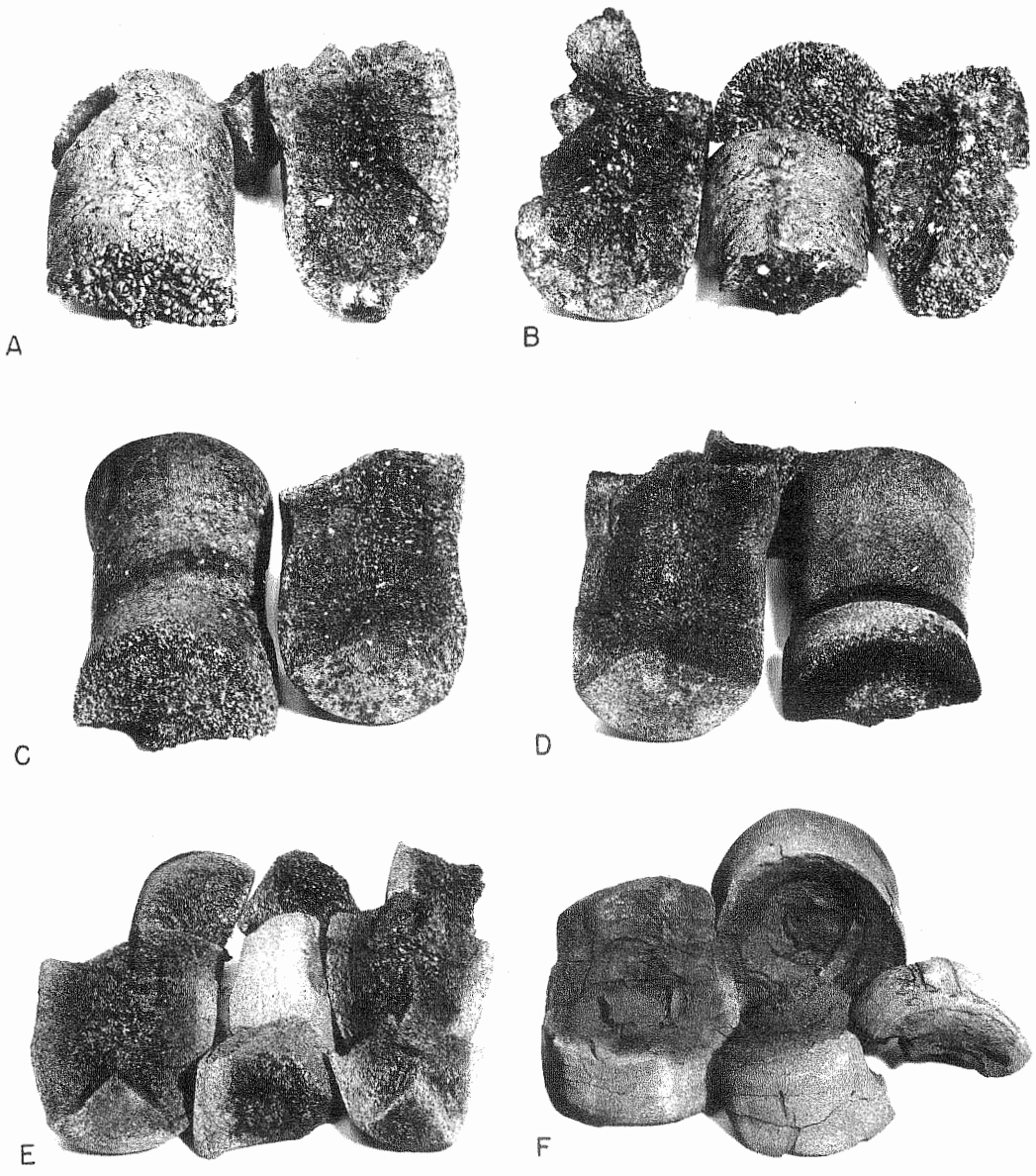
TABLE 17.—No. 6 COAL: INFLUENCE ON COKE OF FUSAIN BLENDED WITH SAMPLES OF STANDARD AND SUB-STANDARD SIZE CONSIST
(Standard coking conditions)

Sample, condition, and blend		Macro-test data of coke			Micro-test data of coke	
		Shatter: % + 1″	Tumbler		Mechanical strength test	
			Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Med. clarain crushed at ⅛″ standard size consist %	−150 Fusain %					
100	0	80.3	81.2	88.2	41.4	0.030
95	5	87.7	77.2	87.2	39.0	0.033
90	10	87.5	70.1	80.6	39.7	0.038
84.2	15.8	83.8	59.4	62.9	37.4	0.030
80	20	53.9	55.0	59.4	35.8	0.030
75	25	36.4	8.4	20.4	30.4	0.021
Med. clarain re-crushed at 1/16″ %	−150 Fusain %					
100	0	73.4	61.3	87.8	41.5	0.042
95	5	89.9	72.3	87.7	40.0	0.045
90	10	88.8	83.3	87.1	39.5	0.040
85	15	88.2	74.3	80.5	35.4	0.044
80	20	72.8	32.5	35.5	31.4	0.040
75	25	53.2	17.9	26.0	33.1	0.036
Med. clarain re-crushed at 1/32″ %	−150 Fusain %					
100	0	86.3	67.2	86.4	42.5	0.033
95	5	76.4	78.6	86.9	38.1	0.038
90	10	89.0	77.8	79.1	36.5	0.030
85	15	89.3	73.6	73.5	30.1	0.031
80	20	73.9	36.4	42.9	29.6	0.033
75	25	67.7	16.9	27.3	28.4	0.031
"Vitrain" crushed at ⅛″ standard size consist %	−150 Fusain %					
100	0	75.3	52.2	83.6	40.6	0.013
95	5	85.0	68.8	88.0	39.5	0.014
90	10	93.9	88.0	90.0	42.2	0.020
85	15	94.3	86.6	87.6	38.8	0.021
80	20	88.6	73.0	78.9	34.8	0.019

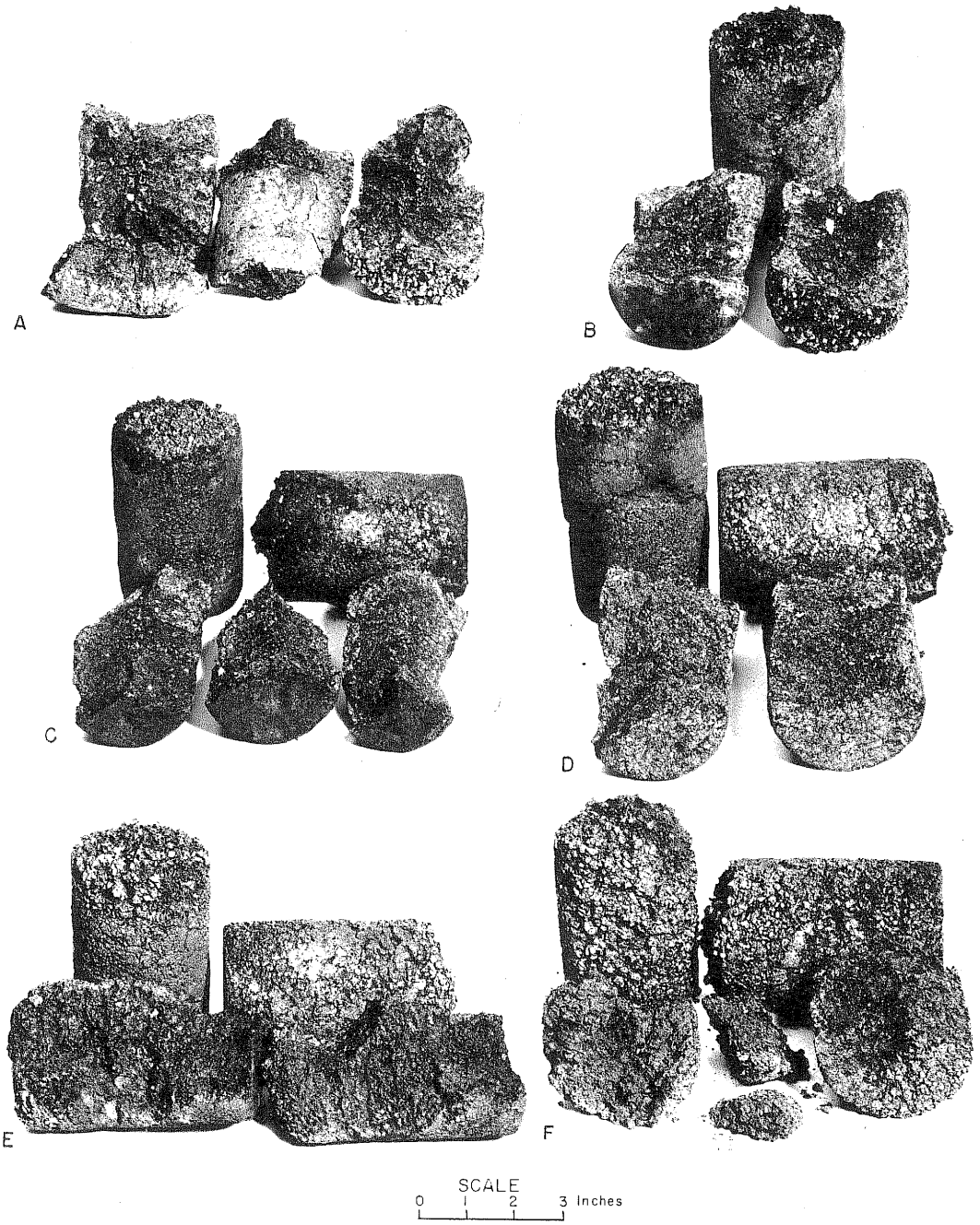
EXPLANATION OF PLATE 2

No. 6 coal: coke produced from different size fractions of medium clarain under standard coking conditions (size fraction in mesh)

A. Coke from 6 x 10 mesh fraction
B. " 10 x 20 "
C. " 20 x 35 "
D. Coke from 35 x 65 mesh fraction
E. " 65 x 150 "
F. " minus 150 "



COKING CHARACTERISTICS OF COAL



COKING CHARACTERISTICS OF COAL

Comparison of the properties of the cokes produced from fusain blended respectively with medium clarain and "vitrain" suggest that there may be an optimum ratio for the proportions of vitrinite and fusinite to produce the strongest coke, provided that constituent size and distribution are adequately controlled. The possibility of blending fine coal from the mines with fusain-rich "fines" from the preparation plants, for the production of high quality coke, is a prospect worthy of serious attention.

The opportunities of utilizing fine coal of high fusain content for blending with the very "bright" coal of the No. 6 seam for the production of cokes of excellent mechanical strength merit further investigation upon pilot plant scale.

Micro-petrographic studies of the cokes of the fusain-coal blends are still in progress, but the initial investigation has revealed that the finely divided fusain and inertinite enters into the coke substance and persists almost in its original condition. Both within the coke material and upon the curved surfaces of the gas cavities, the fusain appears as "aggregate" in the "cement" of other thermally altered coal constituents. Its role appears to be that of "filler" as well as "aggregate," for the macroscopic evaluation of the cokes has confirmed that volumetric changes in the fusain-coal blends appear appreciably less than in the normal coals, thus accounting for their increased stability.

NO. 5 COAL, SALINE COUNTY SEAM AND SAMPLES

As normally developed the No. 5 coal bed is essentially a bright banded coal in which are developed sporadic, thin bands or lenses of clay and shale, together with occasional bodies of calcite, kaolinite, and pyrite that are common along the joint planes (table 18).

The pillar collected for this study represented the full bed section amounting to 91¾ inches.

TABLE 18.—No. 5 COAL: MACRO-PETROGRAPHIC ANALYSIS OF COLUMN

Roof: black to dark gray shale containing vitrain sheets.

Seam Section II (SS II)

8⅛″	Medium to coarse clarain, closely interbedded and rapidly alternating with vitrain sheets up to ¼″ thick and some fusain partings.
⅜″	Fusain horizon with shale and pyrite closely associated.
7½″	Medium to coarse clarain with fine clarain and thin durain intercalations; fusain horizons relatively inconspicuous.
1/16″	Fusain horizon: fine lenticular bodies, partially mineralized.
1¾″	Medium clarain with pyritized joints, a few vitrain sheets up to ⅛″ thick and some small fusain lenticles.
5¼″	Fine to medium clarain.
¾″	Fusain band locally ranging up to 3 inches in other seam profiles with both "soft" and harder varieties of this constituent and minor proportions of vitrain.
5″	Fine clarain to duroclarain with a few vitrain sheets up to ⅛″ thick.
¾″	Vitrain—prominent individual sheet with suggestion of median parting.
5¾″	Fine to medium clarain with fine fusain partings at intervals of approximately 1″; a few vitrain sheets up to ⅛″ thick.
¾″	Vitrain—persistent sheet but of irregular form.
2¼″	Fine to medium clarain with vitrain layers about 1/16″ thick.
¼″	Fusain: typical aggregate of fine lenses with some pyrite and clay.

EXPLANATION OF PLATE 3

No. 6 coal: medium clarain: cokes produced by adding increasing amounts of fusain under standard coking conditions

	Medium clarain percent	Fusain percent		Medium clarain percent	Fusain percent
A.	100	0	D.	85	15
B.	95	5	E.	80	20
C.	90	10	F.	75	25

1″	Coarse to medium clarain with vitrain sheets up to ½″ thick locally.
1″ to ½″	Pyritic concretion horizon with pyritic nodules up to ¼″ in diameter.
1″	Clarain—coarse with persistent vitrain sheets up to ¼″ thick.
2¾″	Clarain—fine to medium with fusain partings.
4″	Medium to coarse clarain with interbanded thin duroclarain and durain. Vitrain sheets up to ⅛″ thick. Fusain partings are common but thin.
11¼″	Fine to medium clarain and duroclarain with thin fusain horizons at intervals of approximately 1″. A few vitrain sheets exceed ⅛″ in thickness.
¼″	Lenticular fusain horizon with imperfectly defined margins; fusain fragments generally flatly lenticular.
1″	Fine to medium clarain and duroclarain.
⅛″	Fusain horizon with markedly lenticular individual fragments.
3¼″	Fine to medium clarain and duroclarain with a few vitrain sheets up to ¼″ thick, rarely greater.
1/16″	Fusain horizon.
4⅝″	Fine to medium clarain, well laminated with occasional coarse vitrains up to ½″ thick. Fusain common both as individual fragments and in thin horizons.
1/16″	Fusain horizon, composed of numerous small lenticular fragments.
1⅞″	Fine to medium clarain with thin vitrain sheets and lenticular fusain particles.
1½″	Medium to coarse clarain with vitrain sheets up to ¼″ thick.
1¾″	Fine to medium clarain with thin vitrain sheets and lenticular fusain.
⅛″	Durain—shale parting with very fine vitrain sheets and fusain lenticles.

Seam Section I (SS I)

3¼″	Fine to medium clarain with vitrain sheets up to 1/16″ thick and fine dispersed fusain lenticles.
⅛″	Clay-shale partings and splint coal in rapid alternation and lateral variation; fine vitrain sheets and lenses occur in all components.
2¾″	Medium to fine clarain with a few vitrain sheets up to ⅛″ and one densely pyritic layer near base.
⅜″	Irregular clay band with vitrain.
⅝″	Clarain with lenticular vitrains and pyritic layers parallel with bedding.
¾″	Carbonaceous shale and argillaceous durain intergrading by abrupt transition. Clay lenticles and flattened silty pellets common. Thin vitrain sheets of highly irregular form.
¼″	Vitrain.
¼″	Fusain rich horizon with shale partings, rounded, "granular" and lenticular vitrain bodies.
1″	Shale or clay band with irregular vitrain sheets and local, flat pyritic lenses, and sheets.
6¾″	Fine, medium to coarse clarain very closely interbedded and rapidly alternating; vitrain sheets up to ⅛″ in thickness.
⅜″	Pyrite and clay band of lenticular character, laterally passing into aggregate of irregular vitrain sheets.
1″	Coarse clarain, with vitrain sheets up to ⅛″ thick, fine fusain lenticles, and clay-shale bodies; pyrite in joint partings.
	Floor: shale with vitrain partings.
91 11/16″	Total thickness in measured column

The lower portion of the pillar, 17¾ inches thick (table 18), contained a high proportion of sedimentary impurity in well defined bands, irregular lenses, and disseminated through the coal, commonly associated with pyrite.

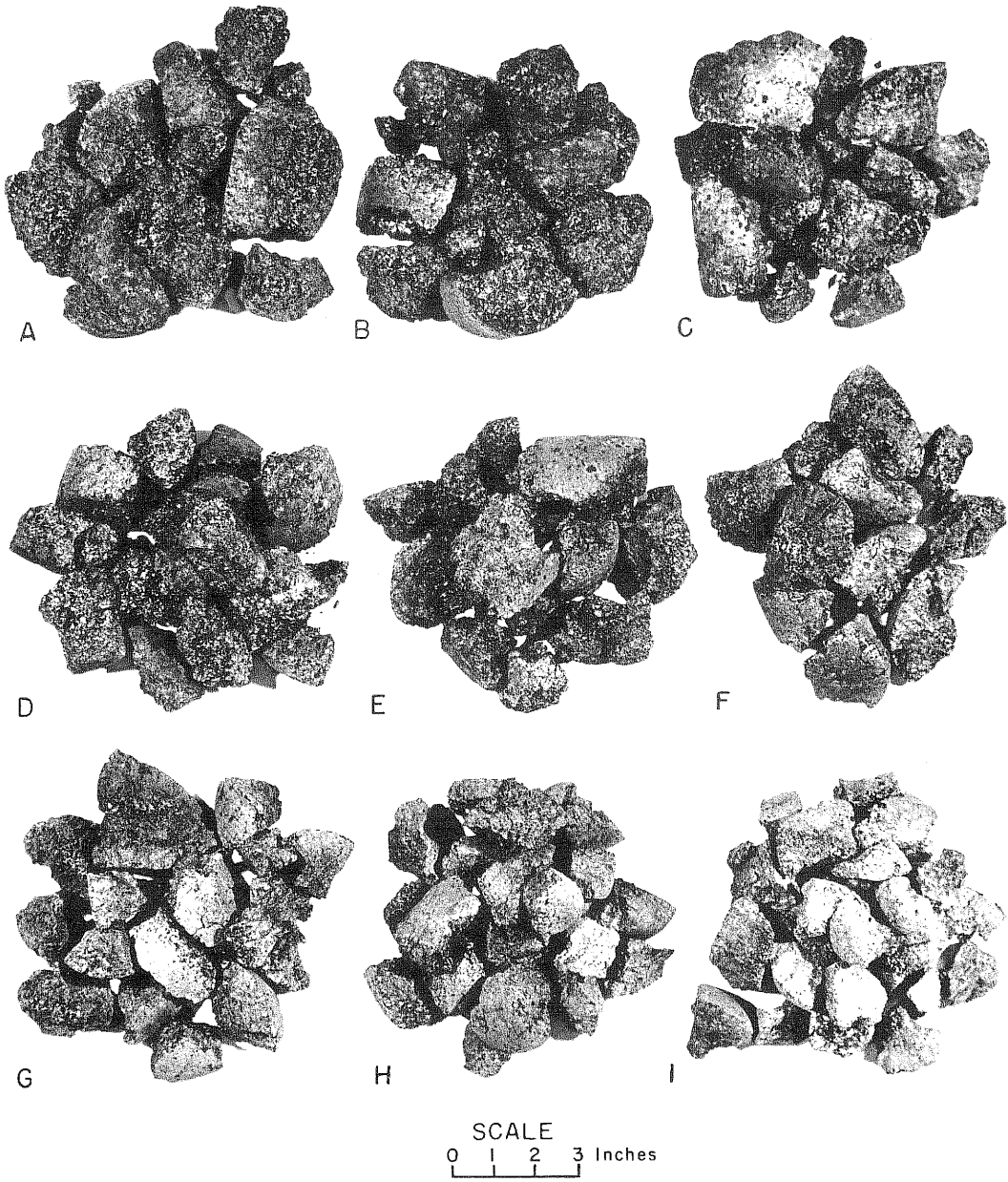
The upper section of the pillar amounted to 74 inches of bright coal in which macrotype variation was not conspicuous. Thin partings of fusain were common and one band of nodular and finely divided pyrite, ½ to 1 inch thick, was prominent about 30 inches from the top. Viewed at low magnification in the polished block, alternations of slightly contrasted coal types were apparent.

In the upper section of the pillar, the variation was principally between dominant medium clarain and much smaller proportions of fine clarain and duroclarain, and rarely, durain; in the lower seam section of the pillar, duller "splinty" bands were more frequent, intimately associated with the sedimentary partings. Individual vitrain bands approached ½ inch in thickness but the

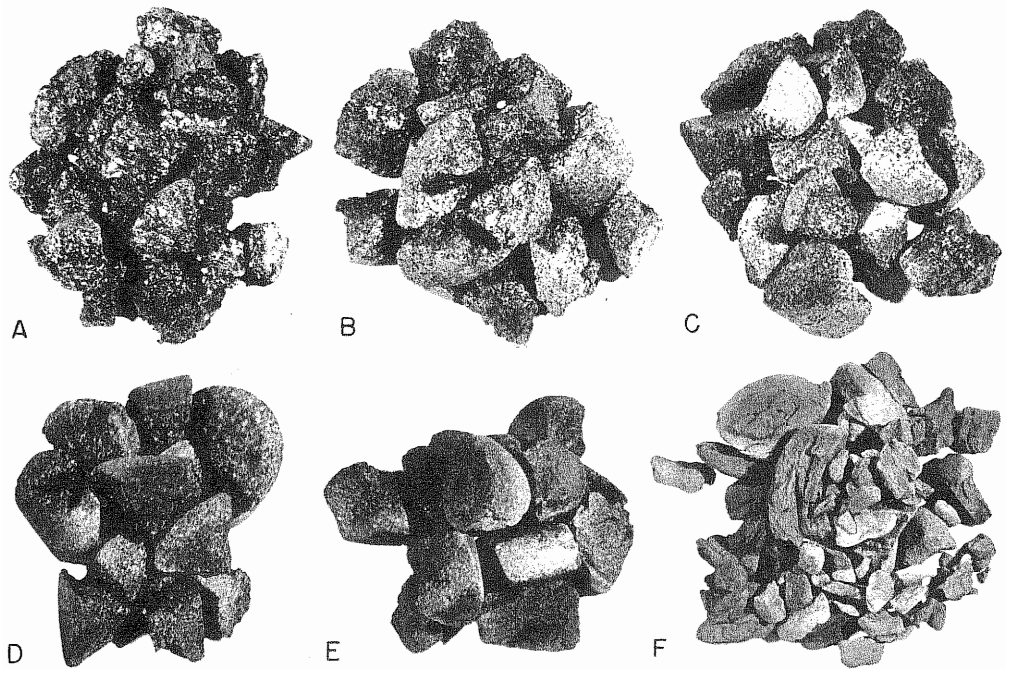
EXPLANATION OF PLATE 4

No. 5 coal: reference pillar: influence of charging temperature on coke under standard coking conditions. After shatter test.

	°F	°C		°F	°C		°F	°C
A.	80	25	D.	600	315	G.	1200	650
B.	200	95	E.	800	430	H.	1400	765
C.	400	205	F.	1000	540	I.	1600	870



COKING CHARACTERISTICS OF COAL



SCALE

0 1 2 3 Inches

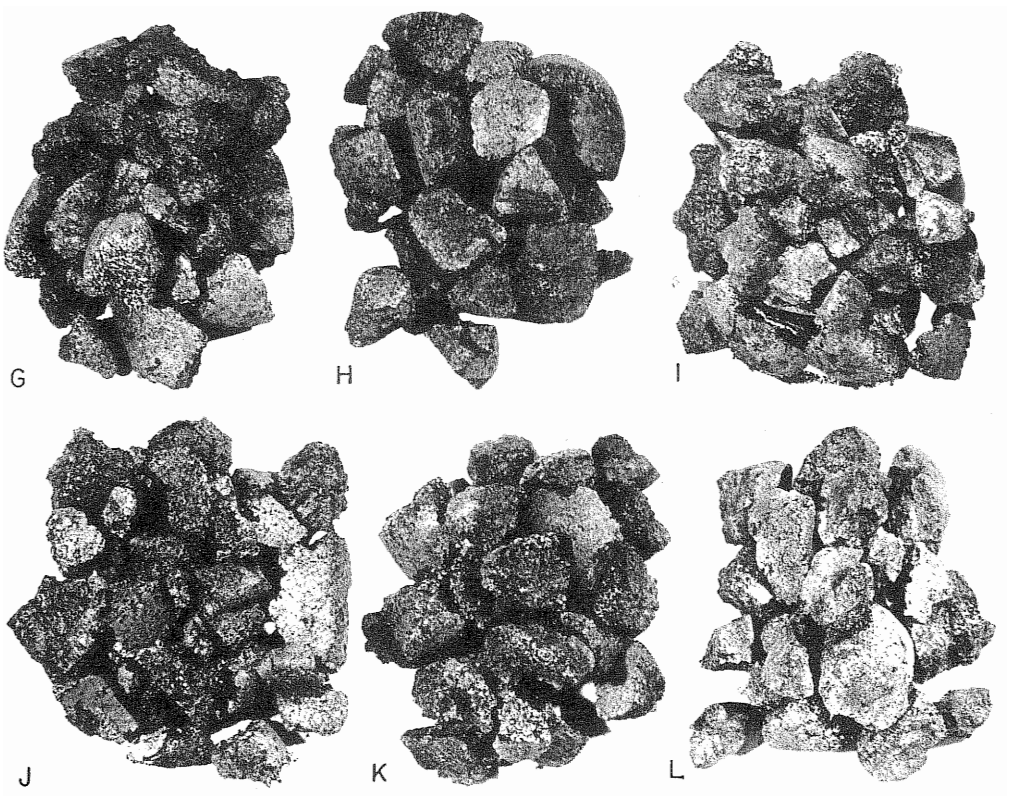


TABLE 19.—No. 5 COAL: SIZE ANALYSIS (CONSIST) OF REFERENCE PILLAR AND SEAM SECTIONS
(Prepared by standard procedure)
Proportion in percentage

Coal size (mesh)	Reference pillar		Seam section I		Seam section II	
	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+6	0.6	0.6	0.1	0.1	0.1	0.1
6 x 10	40.0	40.6	45.9	46.0	47.5	47.6
10 x 20	26.5	67.1	25.9	71.9	26.1	73.7
20 x 35	14.1	81.2	12.9	84.8	12.5	86.2
35 x 65	8.0	89.2	6.8	91.6	6.3	92.5
65 x 150	5.0	94.2	4.0	95.6	3.7	96.2
−150	5.8	100.0	4.4	100.0	3.8	100.0

majority were of the order of 1/8 inch or less.

The generally low degree of differentiation in coal macro-types did not encourage their detailed individual sampling. Consequently, after extracting the column for microscopic examination, three bulk samples were prepared. Of these, the properly proportioned bulk sample of the reference pillar (RP) included both the upper and lower bed sections; the remaining two bulk samples represented the individual lower, high-ash section (SS I) and the upper section of the seam which is normally extracted (SS II).

The bulk samples were prepared initially according to the standard procedure and individual representative sample "units" of approximately 1500 grams weight were stored in sealed cans for the short period between preparation and examination. The size characteristics of each sample varied somewhat more than those of the No. 6 bed (table 19).

PETROGRAPHIC AND CHEMICAL CHARACTERISTICS

In over-all character, both the reference pillar and upper seam section samples were reasonably typical clarains with normal proportions of the common constituents (tables 20A, 20B, fig. 51). The lower section of the seam, contrary to what might have been expected, did

EXPLANATION OF PLATE 5

No. 5 coal reference pillar

I. Coke produced from different size fractions under standard coking conditions. After shatter test.
- A. 6 x 10 mesh size fraction
- B. 10 x 20 mesh size fraction
- C. 20 x 35 mesh size fraction
- D. 35 x 65 mesh size fraction
- E. 65 x 150 mesh size fraction
- F. minus 150-mesh size fraction

II. Coke produced from coal samples with variable size consist, standard coking conditions. After shatter test.
- G. Rebroken at roll setting of 1/16 inch
- H. Rebroken at roll setting of 1/32 inch
- I. Rebroken at roll setting of 1/64 inch
- J. Standard size consist
- K. Burstlein consist
- L. Similar proportions in all size fractions

TABLE 20A.—No. 5 COAL: MICROMETRIC (PETROGRAPHIC) ANALYSES OF MINUS 6-MESH FRACTIONS
(Prepared by standard procedure)

Macrotype or seam sample	Maceral proportions %				Maceral (constituent) mean dimensions, microns				Maceral (constituent) quartile and median dimensions, microns											
									25% quartile				50% quartile (median)				75% quartile			
	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral
Reference pillar	86.2	5.0	4.2	4.5	41.0	4.0	11.1	9.3	5.3	1.7	2.3	2.4	14.5	2.9	4.7	4.8	45.5	5.5	10.5	9.0
Seam section I	75.8	3.5	2.1	18.5	36.5	4.3	15.0	16.5	4.8	1.7	2.9	3.1	11.5	2.9	5.5	6.0	35.8	5.7	14.0	13.4
Seam section II	89.2	3.5	5.5	1.7	54.4	3.6	18.8	8.1	5.7	1.5	2.2	1.9	18.5	2.4	4.5	3.6	61.4	4.3	9.0	6.6
Reference pillar re-crushed at 1/16″	86.4	4.2	5.1	4.2	43.2	3.7	19.7	11.6	5.8	1.6	2.3	2.6	16.0	2.6	5.4	5.0	48.0	4.9	14.0	8.7
Reference pillar re-crushed at 1/32″	87.2	4.2	2.8	5.8	37.8	3.8	12.1	12.9	5.0	1.6	3.3	2.4	15.0	2.8	5.7	4.9	44.0	5.2	9.7	8.7
Reference pillar re-crushed at 1/64″	84.4	4.0	6.3	5.4	28.2	3.4	10.9	11.3	4.8	1.6	2.5	2.3	11.0	2.5	5.1	4.6	30.0	4.8	9.6	9.0
Fusain −150 mesh	15.0	1.2	81.1	2.7																

TABLE 20B.—No. 5 COAL: MICROMETRIC (PETROGRAPHIC) ANALYSES OF INDIVIDUAL SIZE FRACTIONS OF STANDARD SAMPLES

Seam sample (mesh)	Maceral proportions %				Maceral (constituent) mean dimensions, microns				Maceral (constituent) quartile and median dimensions, microns											
									25% quartile				50% quartile (median)				75% quartile			
	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral	Vitrinite	Exinite	Inertinite	Mineral
Reference pillar																				
+6	60.51	10.64	6.55	22.29	27.33	7.86	23.56	25.63	6.4	2.6	2.8	5.4	10.2	3.7	4.8	7.5	31.0	6.8	15.0	23.0
6 x 10	85.11	6.25	3.21	5.43	37.79	3.83	7.94	10.00	5 0	1.7	2.0	2.5	14.0	2 9	4.0	4 8	44.0	5.6	8.4	8.9
10 x 20	88.70	4.03	4.05	3.22	51.75	4.07	12.25	7.00	5.1	1.7	2.1	2.2	14.5	2.8	4.6	4.3	50.0	5.4	10.0	7.5
20 x 35	89.00	4.23	2.91	3.86	45.32	3.80	15.77	9.31	5.7	1.6	2.5	2.3	16.0	2.6	5.6	4.8	50.0	5.1	15.0	9.2
35 x 65	86.91	4.96	4.71	3.42	39.16	4.27	14.60	10.24	5.8	1.7	2.5	2.1	17.0	3.1	5.2	4.2	49.0	5.8	10.0	8.9
65 x 150	86.56	4.45	4.31	4.68	31.49	4.32	14.77	15.16	6.8	1.7	3.6	3.8	17.0	3.1	7.6	7.2	44.0	5.9	20.0	17.0
−150	78.61	3.19	13.45	4.75	16.73	4.45	7.99	7.43	4.7	1.9	2.8	2.6	9.2	3.5	5.4	4.9	22.5	6.3	9.2	8.4
Seam section I																				
6 x 10	75.03	3.89	0.48	20.60	40.07	4.62	5.23	18.68	4.6	1.8	2.9	2.6	11.0	3.1	4 8	5.6	38.0	6.1	7.6	13.0
10 x 20	73.29	3.45	3.35	19.91	34.34	4.24	31.81	16.16	4.9	1.7	3.5	3.7	11.5	3.0	7.2	6.6	30 0	5.9	25.0	14.0
20 x 35	81.26	3.07	3.40	12.27	40.65	3.77	19.07	12.81	5.2	1.6	2.4	3.5	13.5	2.5	5.8	6.5	42.0	4.6	18 0	14.0
35 x 65	80.01	2.53	2.82	14.64	34.76	3.25	14.78	14.35	4.6	1.5	1.8	3.2	12.0	2.4	3.5	6.0	44.0	4 4	8.7	13.0
65 x 150	76.11	3.68	3.34	16.87	26.55	4.53	15.39	16.04	5.6	1.8	4.5	3.5	14.5	3.1	7.4	7.0	36.0	6 0	20 0	17.0
−150	77.68	3.39	5.54	13.39	12.36	3.88	6.78	9.56	3.7	1.7	1.9	2.4	7.1	2.9	3.6	5.1	16.0	5.5	7.6	10.0
Seam section II																				
6 x 10	90.57	3.65	4.54	1.24	65.41	3.86	24.75	7.30	5.8	1.5	2.2	1.9	20.0	2.4	4.4	3.6	70.0	4.5	8.8	6.1
10 x 20	86.85	3.48	6.75	2.92	48.54	3.27	16.10	11.77	5.5	1.5	2.3	2.1	16.0	2.2	4.6	4.0	54.0	3 9	8.7	7.7
20 x 35	90.69	3.08	4.66	1.57	50.09	3.13	11.39	6.67	6.2	1.5	2.0	1.7	22.0	2.3	4.1	2.8	66.0	4 0	8.8	6.0
35 x 65	92.14	3.35	3.13	1.38	42.81	3.27	10.82	5.20	6.3	1.6	2.1	2.3	18.5	2.4	4.8	4.4	55 0	4 3	12.0	7.5
65 x 150	90.62	3.81	4.24	1.33	29.28	3.64	11.25	5.46	5.6	1.6	2.3	1 9	15.0	2.6	4.8	3.5	42.0	5.1	10 0	6.3
−150	79.37	2.34	16.40	1.89	15.34	3.65	7.64	6.31	4.5	1.7	2.6	1.8	8.9	2.8	4.9	3.2	21.0	5.4	8.5	6.6

not exhibit increased proportions of either inertinite or exinite; as compared with the reference pillar and upper section of the seam the vitrinite and inertinite content were lower and the mineral matter was greater. The contrast between the lower and upper sections of the bed could not be related specifically to variation in coal type (that is, to substantial changes in relative proportions of the macerals) but rather to increased proportions of sedimentary mineral matter in what would otherwise have been normal clarain. The relative proportions of exinite in the reference pillar and two seam sections (table 20A) is apparently anomalous and may be due to error in sampling.

In chemical composition (table 21), carbon content varied sympathetically with both vitrinite and inertinite content, but it is more than probable that the latter was the controlling entity (fig. 51). Ash, mineral matter, and sulfur all showed broadly sympathetic variation. Neither volatile content nor hydrogen varied significantly.

Gieseler values and free swelling indices were determined upon the three bed standard samples (RP, SS I, SS II), as well as fluidity determinations and free swelling tests using modified procedure in which the sample was not crushed to accepted test specifications (table 21, fig. 52). It is not known to what extent the differences exhibited by the two sets of fluidity data may be due to purely mechanical causes inherent in the different particle size of the samples, nevertheless they are of interest. Softening temperatures were lower in the samples using accepted test procedures although not significantly so in seam section II, whereas setting temperatures were very similar as determined by both techniques. The samples tested with "original" size as obtained by sieving exhibited slightly lower temperatures of maximum fluidity than samples with Gieseler test preparation and, much greater maximum fluidity (dial divisions per minute). For both the reference pillar and seam section II, the "original" samples had a lower free swelling index than the samples of standard FSI test preparation.

In the various particle size fractions of the samples prepared by standard procedure, volumetric proportions and constituent (maceral) width varied systematically (tables 20A, 20B; figs. 53, 55, 56).

In samples of the reference pillar, the vitrinite content was low in the small plus 6-mesh fraction, rose greatly in the next size range (6 x 10 mesh) and thereafter increased slightly to a maximum in the 20 x 35 mesh particle size group and diminished most rapidly in the minus 150-mesh portion of the sample. The quantity of inertinite increased in the minus 150-mesh fraction.

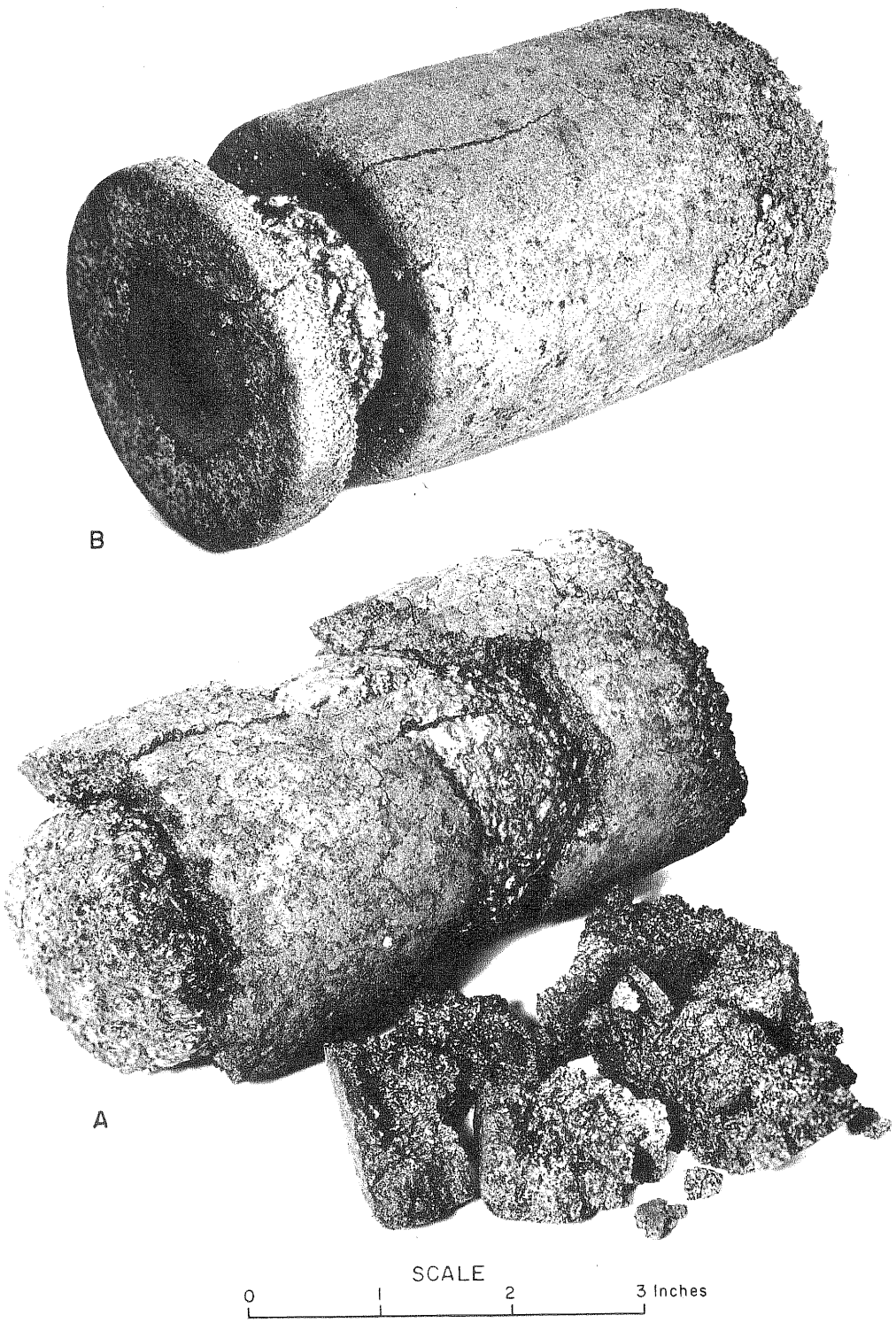
The average and median maceral dimensions in the various size ranges of the reference pillar likewise presented a systematic variation (table 20B, fig. 56). Disregarding the insignificant plus 6-mesh fraction, for vitrinite the average size increased to a maximum in the 10 x 20 mesh range while those of the

EXPLANATION OF PLATE 6

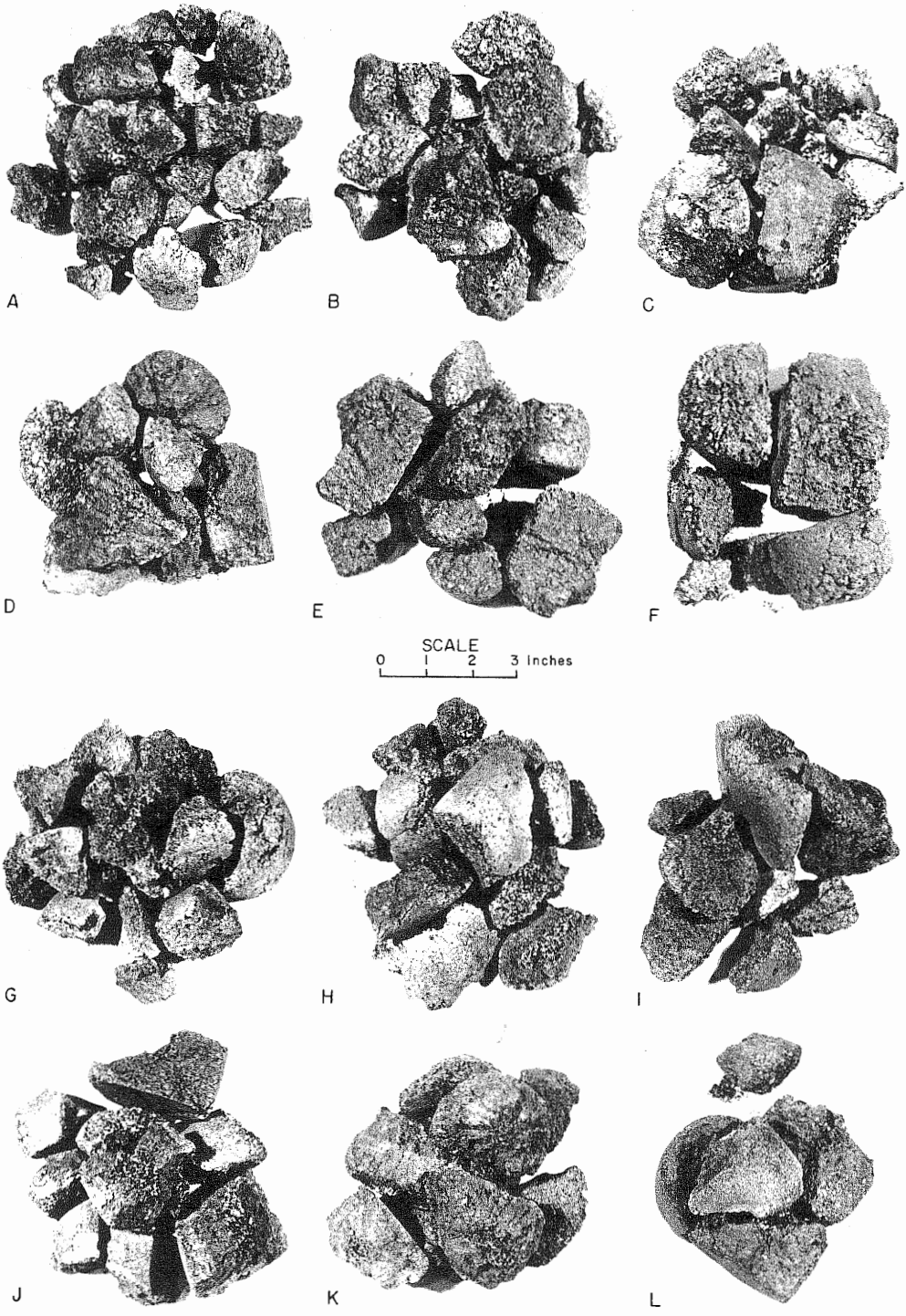
No. 5 coal reference pillar: extrusion coke

A. Extrusion coke produced under standard coking conditions

B. Extrusion coke from coal recrushed at 1/16 inch plus 10 percent fusain and produced under standard coking conditions



COKING CHARACTERISTICS OF COAL


A
B
C
D
E
F
SCALE
0 1 2 3 Inches
G
H
I
J
K
L

other three recorded macerals generally decreased in the larger size fractions. Median dimensions for vitrinite increased throughout the remaining fractions of diminishing size with a sharp decline in the finest fraction (minus 150-mesh). For inertinite and mineral matter, except for the plus 6-mesh fraction, the median dimension increased throughout the plus 150-mesh sieve ranges to a pronounced maximum in the 65 x 150 mesh fraction; in the minus 150-mesh portion, their dimensions closely corresponded with their general average. Exinite exhibited a gradual decline in median sizes to a minimum in the 20 x 35 mesh range and thereafter increased slowly into the finest fraction.

Volumetric proportions of the macerals present in seam section I did not vary greatly among the various size fractions (table 20B, fig. 57). Vitrinite reached maximum proportions in the size range 20 x 35 mesh with mineral matter correspondingly lower; inertinite was least concentrated in the 6 x 10 mesh fraction, increased slightly thereafter, and attained a modest maximum in the finest fraction (minus 150-mesh).

In the particle size fractions of seam section I, not only were the volumetric proportions of vitrinite generally low (figs. 57, 58), but the average and median widths were reduced (table 20B, fig. 58) as compared with these values for the reference pillar samples. After initial fluctuation the average width of the vitrinite decreased rapidly while the median values increased throughout the plus 150-mesh material; they were much reduced in the minus 150-mesh fraction. Average and median values for inertinite were higher in the 10 x 20 and 65 x 150 mesh fractions, and much lower in the minus 150-mesh range. Exinite average and median widths were at a minimum in the 35 x 65 mesh fraction. For the mineral matter, average component sizes fluctuated with greatest values in the 65 x 150 mesh fraction; the median width varied slightly but generally increased into the 65 x 150 mesh fraction.

Seam section II, the conspicuously brilliant bench of the seam which is normally worked, is characterized by a

EXPLANATION OF PLATE 7

No. 5 coal, fusain blends

I. Cokes produced by adding increasing amounts of minus 150-mesh fusain to the standard reference pillar (standard breakage at 1/8 inch roll setting); standard coking conditions. After shatter test.

	Reference pillar percent	Fusain percent		Reference pillar percent	Fusain percent
A.	100	0	D.	85	15
B.	95	5	E.	80	20
C.	90	10	F.	75	25

II. Cokes produced by adding increasing amounts of minus 150-mesh fusain to the reference pillar (rebroken at 1/16 inch roll setting); standard coking conditions. After shatter test.

	Reference pillar percent	Fusain percent		Reference pillar percent	Fusain percent
G.	100	0	J.	85	15
H.	95	5	K.	80	20
I.	90	10	L.	75	25

high volumetric proportion of vitrinite which in the prepared samples reached a maximum in the 35 x 65 mesh fraction, and declined greatly in the minus 150-mesh material (table 20B, fig. 59). A subordinate minimum in the 10 x 20 mesh range was accompanied by an increase in the proportions of inertinite. With the exception of the inertinite which increased substantially in the finest material, the other seam constituents varied but slightly between the different size fractions of the standard sample.

The maceral widths in seam section II were also a little unusual (table 20B, fig. 60). The average maceral width of the greatly dominant vitrinite decreased precipitously into the finest fractions with one reversal in the 20 x 35 mesh range. After an increase from the 10 x 20 mesh into the 20 x 35 mesh the median width also decreased sharply, possibly a response to the higher proportions of vitrinite present in substantially coarser bands which suffered progressive pulverization. Except for a minor departure from the trend in the 65 x 150 mesh range the average width of inertinite decreased progressively with particle size; the median width increased generally throughout the diminishing size fractions. Exinite exhibited little variation in either average or median values but retained the feature of slightly increased dimensions in the coarsest and finest fractions.

When broken alone, the upper seam section coal (SS II) exhibited a progressive degradation of the strongly dominant vitrinite in the medium and lower particle size fractions, an effect apparently due to the lesser proportions of microclastic coal bands, particularly fine clarain.

As a further check upon variation in character of the prepared sample, chemical analyses were made of each size fraction. Fluidity and free swelling values were also obtained by the modification of accepted test methods using the material in the original sieved sample size instead of crushing to the prepared size of the accepted test procedure (tables 20B, 22; figs. 53, 54). Generally, carbon content varied sympathetically with inertinite (highest in plus 6- and minus 150-mesh fractions) and antithetically with vitrinite. Ash increased progressively with the de-

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EXPLANATION OF PLATE 8

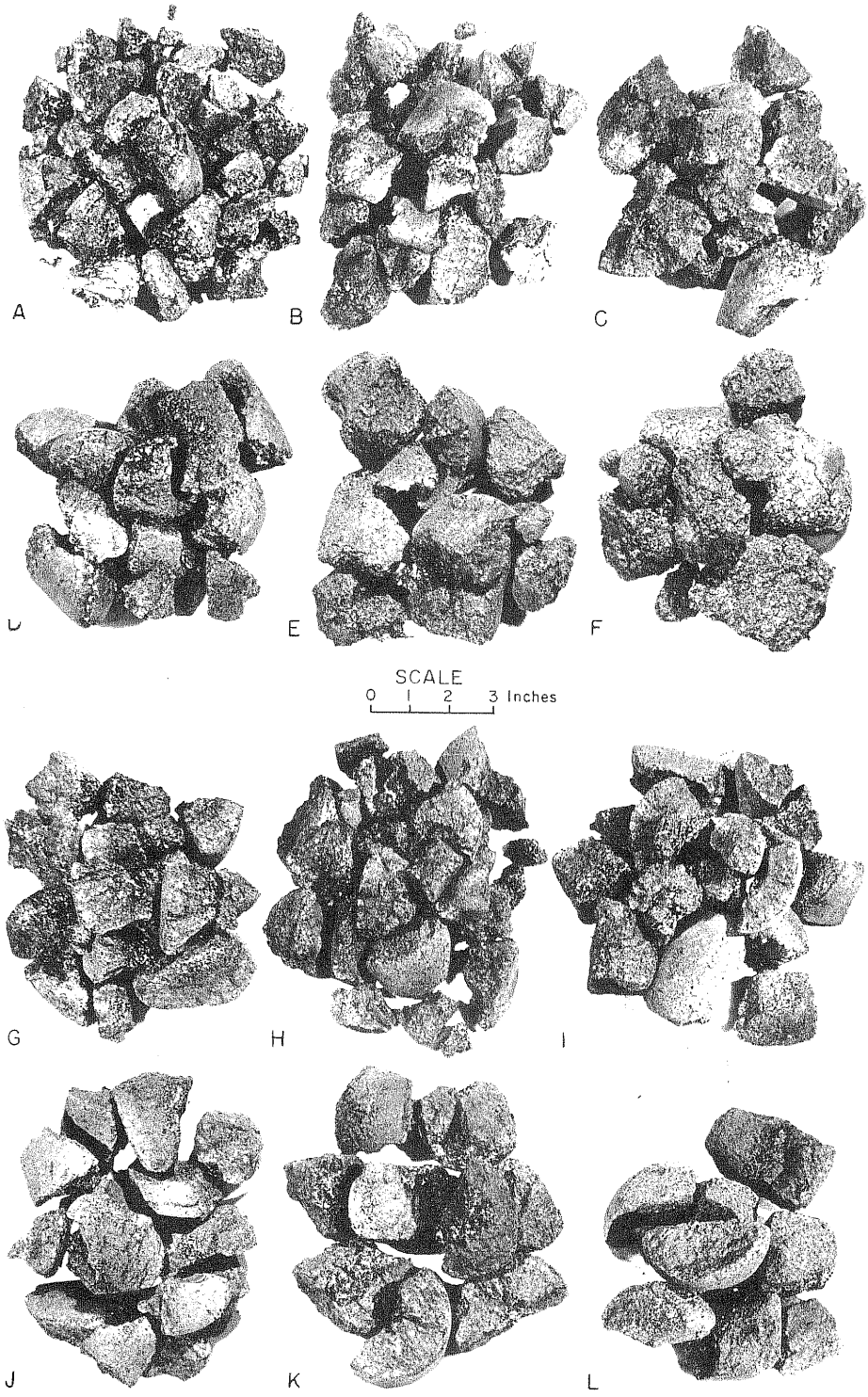
No. 5 coal, fusain blends

I. Cokes produced by adding increasing amounts of minus 150-mesh fusain to the seam section II (standard breakage at 1/8 inch roll setting); standard coking conditions. After shatter test.

	Seam section II percent	Fusain percent		Seam section II percent	Fusain percent
A.	100	0	D.	85	15
B.	95	5	E.	80	20
C.	90	10	F.	75	25

II. Cokes produced by adding increasing amount of minus 150-mesh fusain to the seam section II (rebroken at 1/16 inch roll setting); standard coking conditions. After shatter test.

	Seam section II percent	Fusain percent		Seam section II percent	Fusain percent
G.	100	0	J.	85	15
H.	95	5	K.	80	20
I.	90	10	L.	75	25


A
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SCALE
0 1 2 3 Inches
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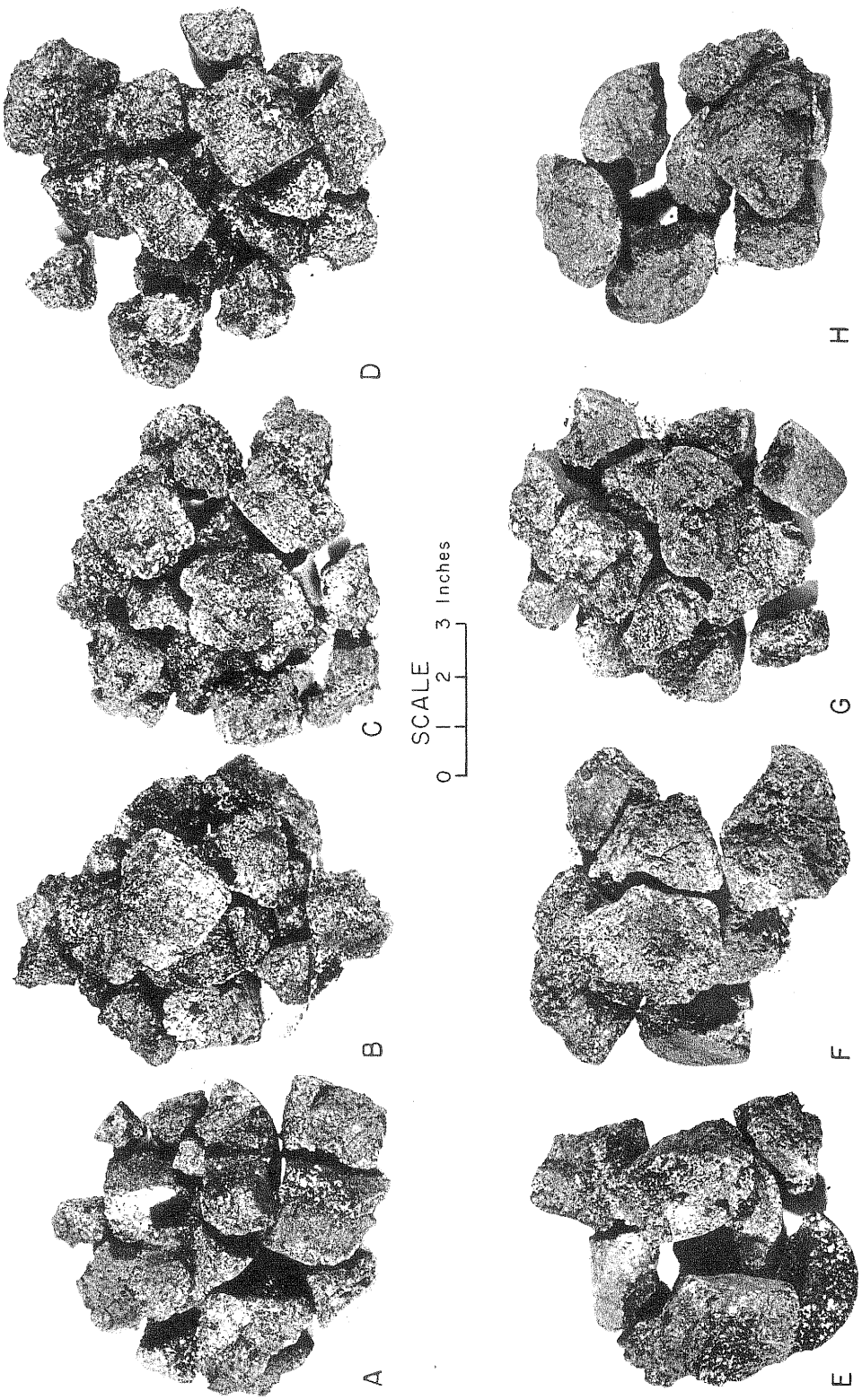
A
B
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SCALE
0 1 2 3 Inches
E
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G
H

crease in particle dimensions. Volatile matter, hydrogen and nitrogen decreased slightly but progressively throughout the range from coarsest to finest material.

The Gieseler values for softening temperature decreased slightly at first with particle size, but tended to increase, although erratically, in the three finer fraction ranges (below 20 x 35 mesh); the setting temperature generally decreased with reduction in particle size (table 22, fig. 54). As between the original (modified fluidity test) and prepared (Gieseler test) samples, the former almost invariably exhibited higher softening and lower setting temperatures, thus reducing the effective plastic range. The temperatures of maximum fluidity were similar as determined by both procedures but Gieseler (prepared) values were generally slightly higher. Maximum fluidity temperature declined in both tests in the finer sizes. In both series of tests the maximum fluidity (dial divisions per minute) declined rapidly from the larger sizes down to the 20 x 35 mesh size fractions, then remained reasonably constant into the minus 150-mesh material. The free swelling indices declined generally from coarse to fine material in both test series. Results were somewhat more erratic in the modified test upon the original samples.

Although certain comparisons are possible between the plastic properties of the samples and their petrographic character (that is, softening and setting temperatures relative to vitrinite content), variation appears to have been largely anomalous and probably was due more to inevitable variations in experimental conditions than to inherent characteristics of the fractions.

COKING STUDIES

Factors in the Coking Cycle

Subject to the same limitations of time and equipment as was true of No. 6 coal, the coking properties of the reference pillar samples of the No. 5 coal, as influenced by factors in the heating cycle, were submitted to "standard" experimental procedures almost identical with those used in the study of the No. 6 coal. These coking tests were pre-

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EXPLANATION OF PLATE 9

No. 6 coal, "resin" additive and "resin" additive plus fusain

I. Cokes produced by adding increasing amounts of "resin" to the special representative sample; standard coking conditions. After shatter test.

	Coal percent	"Resin" percent		Coal percent	"Resin" percent
A.	100	0	C.	95	5
B.	97½	2½	D.	92½	7½

II. Cokes produced by adding "resin" and fusain to the special representative sample: standard coking conditions. After shatter test.

	Coal percent	Fusain percent	"Resin" percent		Coal percent	Fusain percent	"Resin" percent
E.	90	10	5	G.	90	10	2½
F.	80	20	5	H.	80	20	2½

TABLE 21.—No. 5 COAL: CHEMICAL ANALYSES AND FLUIDITY AND SWELLING DATA OF PLUS 6- AND MINUS 6-MESH SAMPLES
(Standard crushing procedure)

Sample, fraction and/or condition	As received				Moisture and ash free							Plasticity data					Swelling index
	Proximate				Proximate		Ultimate										
	Moist.	Ash	Vol.	F. C.	Vol.	F. C.	H	C	N	O	S	Btu/lb.	Soft. temp. °C	Fusion temp. °C	Max. fluid temp. °C	Setting temp. °C	Max. fluid div/min.	
Reference pillar																		
Prepared*	5.2	10.9	34.4	49.5	41.0	59.0	5.46	82.34	1.91	8.72	1.57	14548	388	412	433	464	57	5½
Original†													398	416	432	463	136	3½
Reference pillar +6 mesh																		
Prepared	5.2	9.6	35.3	49.9	41.4	58.6	5.53	82.78	1.95	8.41	1.33	14601	391	416	432	465	57	5
Original													392	409	428	462	149	3½
Seam section I +6 mesh																		
Prepared	4.6	31.0	26.9	37.5	41.8	58.2	5.48	78.80	1.89	9.13	4.70	14262	412	—	427	453	1	1
Original													408	—	422	450	2	3
Seam section I −6 mesh																		
Prepared	4.8	26.2	27.8	41.2	40.3	59.7	5.52	80.09	1.87	8.56	3.96	14254	400	—	432	453	2	2½
Original													414	—	430	454	2	3
Seam section II +6 mesh																		
Prepared	5.3	6.6	36.0	52.1	40.9	59.1	5.53	83.28	1.94	8.46	0.79	14657	390	413	431	464	78	5½
Original													401	411	433	457	151	3½
Seam section II −6 mesh																		
Prepared	5.4	5.5	36.0	53.1	40.4	59.6	5.51	83.05	1.95	8.70	0.79	14676	391	420	437	461	16	5½
Original													393	411	433	462	280	3½

*Prepared—Gieseler plasticity data and free swelling index.
†Original—Fluidity and swelling index obtained by modified procedure.

TABLE 22.—No. 5 COAL: CHEMICAL ANALYSES AND FLUIDITY AND SWELLING DATA OF REFERENCE PILLAR SAMPLES AND SIZE FRACTIONS
(Produced by standard procedures)

Sample, fraction and/or condition	As received				Moisture and ash free								Plasticity data					Swelling index
	Proximate				Proximate		Ultimate						Soft. temp. °C	Fusion temp. °C	Max. fluid temp. °C	Setting temp. °C	Max. fluid div/min.	
	Moist.	Ash	Vol.	F. C.	Vol.	F. C.	H	C	N	O	S	Btu/lb.						
Entire reference pillar																		
Prepared*	5.2	10.9	34.4	49.5	41.0	59.0	5.46	82.34	1.91	8.72	1.57	14548	388	412	433	464	57	5½
Original†													398	416	432	463	136	3½
Reference pillar + 6 mesh																		
Prepared	5.2	9.6	35.3	49.9	41.4	58.6	5.53	82.78	1.95	8.41	1.33	14601	391	416	432	465	57	5
Original													392	409	428	462	149	3½
Reference pillar 6 x 10 mesh																		
Prepared	5.1	10.5	34.7	49.7	41.1	58.9	5.53	82.55	1.95	8.38	1.59	14569	390	414	432	461	29	5
Original													403	414	428	456	38	3½
Reference pillar 10 x 20 mesh																		
Prepared	5.0	10.5	34.4	50.1	40.7	59.3	5.48	82.14	1.93	8.94	1.51	14583	390	415	431	457	25	5½
Original													401	414	430	456	36	3
Reference pillar 20 x 35 mesh																		
Prepared	4.8	11.1	34.0	50.1	40.4	59.6	5.46	82.13	1.90	8.97	1.54	14423	388	—	436	461	5	5
Original													394	420	427	458	7	3
Reference pillar 35 x 65 mesh																		
Prepared	4.9	11.4	32.5	51.2	38.8	61.2	5.45	81.95	1.91	9.09	1.60	14458	392	—	427	458	3.5	4½
Original													392	421	428	457	6	2½
Reference pillar 65 x 150 mesh																		
Prepared	5.0	11.4	32.5	51.1	38.9	61.1	5.42	81.90	1.91	9.16	1.61	14447	387	—	425	460	5	4
Original													387	—	422	460	4	3½
Reference pillar −150 mesh																		
Prepared	4.3	13.7	29.6	52.4	36.1	63.9	5.06	83.08	1.71	8.47	1.68	14496	397	—	421	454	2	2
Original													no resistance to movement					1½

*Prepared—Gieseler plasticity data and free swelling index.
†Original—Fluidity and swelling index obtained by modified procedure.

TABLE 23.—No. 5 Coal: Influence of Charging Temperature on Coke
(Reference pillar, standard size consist, rate of temperature increase, final coking temperature, final coking period 2 hours)

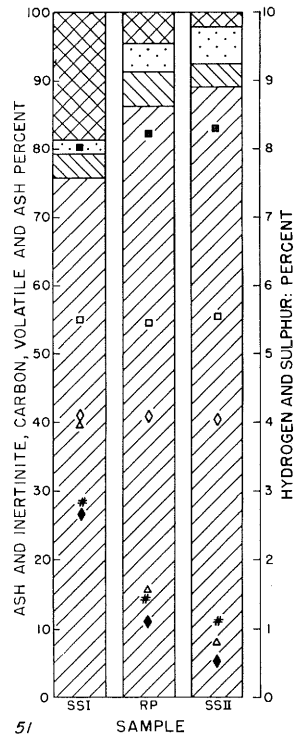
Oven temp. on charging		Macro-test data of cokes									Micro-test data of cokes	
		Shatter			Tumbler						Mechanical strength test	
		% + 1″			Stability: % + 1″			Hardness: % + ¼″			% + 65m	Ratio 28/65m
°C±	°F	Initial	Inter	Final	Initial	Inter	Final	Initial	Inter	Final		
25	80	97.6	86.0	82.7	96.8	82.4	72.2	97.4	89.2	84.6	48.9	0.054
95	200	98.2	87.7	83.0	98.1	89.7	77.3	98.1	90.8	84.6	48.6	0.039
95*	200	96.0	77.7	68.1	98.1	68.9	55.2	98.1	88.2	82.4	—	—
205	400	97.9	83.0	78.2	98.2	81.4	73.6	98.2	90.0	85.4	46.7	0.038
315	600	99.1	84.8	74.7	99.1	82.6	72.9	99.1	89.5	83.9	48.0	0.032
430	800	99.4	78.4	76.7	99.4	85.8	73.8	99.4	90.0	83.7	47.5	0.037
540†	1000	99.6	85.5	81.2	99.2	78.6	70.3	99.3	94.6	84.6	46.9	0.044
540*	1000	99.7	70.7	53.1	99.8	69.4	56.2	99.8	91.6	85.4	—	—
650	1200	99.6	84.4	74.3	99.6	72.0	48.4	99.6	90.2	83.8	46.8	0.040
650*	1200	98.9	83.1	66.3	95.6	56.6	36.9	99.4	90.1	83.8	—	—
765	1400	94.5	80.3	59.0	99.2	69.9	52.1	99.2	90.3	84.5	49.8	0.052
870	1600	94.8	55.4	41.0	99.0	57.9	46.4	99.0	91.0	86.2	52.1	0.084

*Extrusion plug.
†Temperature selected as standard for No. 5 coal, results are mean of several runs.

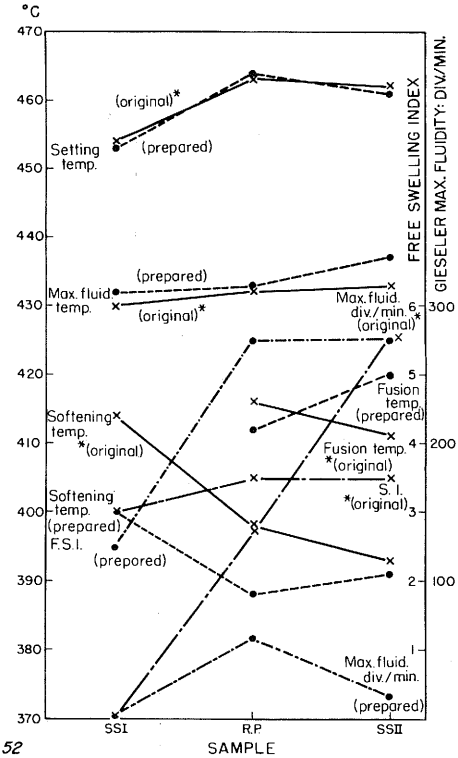
TABLE 24.—No. 5 COAL: PROXIMATE ANALYSIS OF COKE SAMPLES PRODUCED BY VARYING CHARGING TEMPERATURE
(Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period)

Charging temperature of oven		Air dried							Moisture and ash free			
°C±	°F	Coke yield %*	Moist.	Ash	Vol.	F. C.	S	Btu/lb.	Vol.	F. C.	S	Btu/lb.
25	80	75	2.0	17.2	2.3	78.5	1.21	—	2.8	97.2	1.50	—
95	200	74	1.9	17.5	2.2	78.4	1.21	—	2.7	97.3	1.50	—
205	400	75	1.6	17.5	2.1	78.8	1.20	11609	2.6	97.4	1.48	14350
315	600	72	1.7	17.2	2.1	79.0	1.20	—	2.6	97.4	1.48	—
430	800	71	1.4	17.0	1.8	79.8	1.17	—	2.2	97.8	1.43	—
540	1000	71	1.6	17.5	1.9	79.0	1.22	—	2.3	97.7	1.51	—
650	1200	70	0.9	16.6	2.1	80.4	1.24	—	2.5	97.5	1.50	—
765	1400	66	0.8	16.9	1.7	80.6	1.25	—	2.1	97.9	1.52	—
870	1600	65	0.7	17.0	1.3	81.0	1.20	—	1.6	98.4	1.46	—

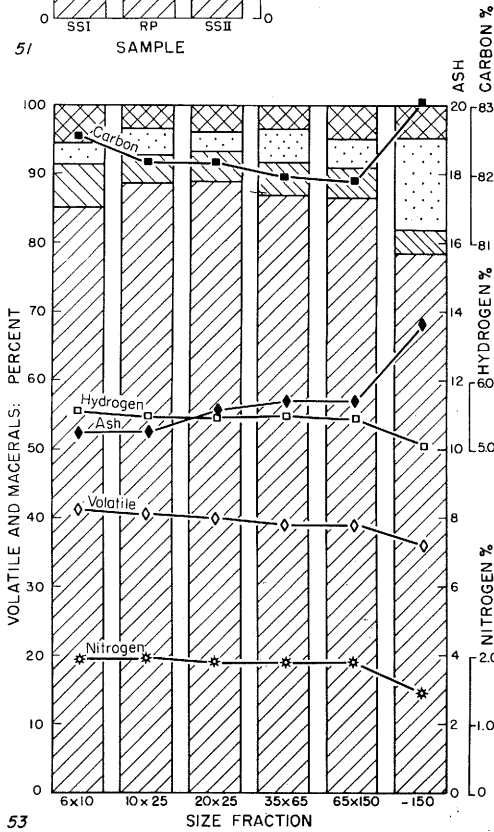
*Average of three test samples, not adjusted for moisture.


ASH AND INERTINITE, CARBON, VOLATILE AND ASH PERCENT
HYDROGEN AND SULPHUR: PERCENT
SSI
RP
SSII
SAMPLE
KEY
Carbon
Hydrogen
Volatile
Sulphur
Ash
Nitrogen
Ash and Inertinite
Moisture
Vitrinite
Exinite
Inertinite
Mineral Matter

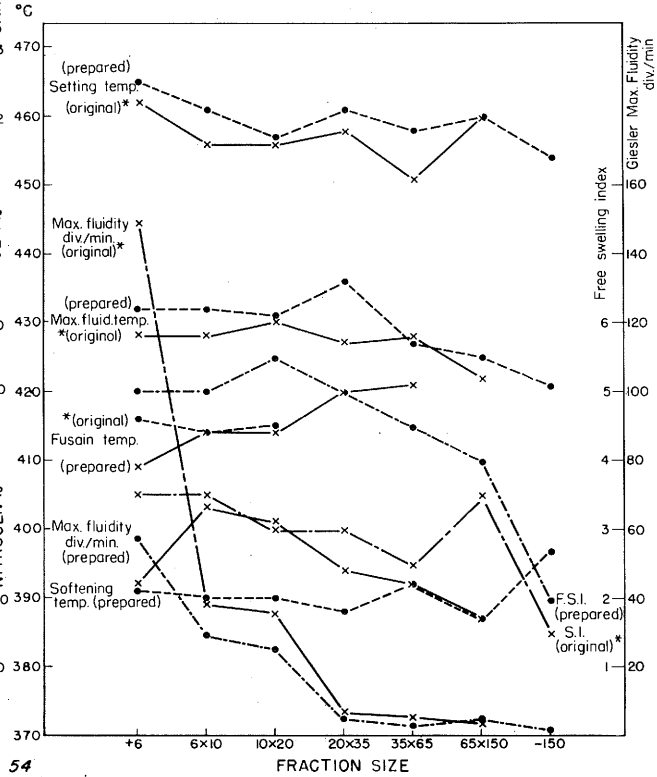
51


°C
FREE SWELLING INDEX
GIESELER MAX. FLUIDITY: DIV./MIN.
(original)*
(prepared)
Setting temp.
Max. fluid temp.
Max. fluid. div./min.
Softening temp.
Fusion temp.
S.I.
F.S.I.
SSI
R.P.
SSII
SAMPLE

52


VOLATILE AND MACERALS: PERCENT
ASH
CARBON %
HYDROGEN %
NITROGEN %
Carbon
Hydrogen
Ash
Volatile
Nitrogen
6x10
10x25
20x25
35x65
65x150
-150
SIZE FRACTION

53


°C
Giesler Max. Fluidity div./min
Free swelling index
(prepared) Setting temp.
(original)*
Max. fluidity div./min. (original)*
(prepared) Max. fluid.temp. *(originol)
*(original) Fusain temp. (prepared)
Max. fluidity div./min. (prepared)
Softening temp. (prepared)
F.S.I. (prepared)
S.I. (original)*
+6
6x10
10x20
20x35
35x65
65x150
-150
FRACTION SIZE

54

ceded by three initial sets of tests to establish "standard" coking procedure as follows:

Effect of Charging Temperature.—Retorts containing standard charges of the reference pillar sample were introduced into the oven at various temperatures ranging from 25°C to 870°C. Thereafter a standard rate of temperature increase of 3.6° per minute was continued up to a final coking temperature of 1010°C, which was maintained for a period of two hours. In this first exploration series, final temperature and time were provisional standards only, to be adjusted, if desirable, in accordance with the results of later studies.

As in the case of the No. 6 coal, temperature of charging appeared to be significant in relation to characteristics of the coke produced, as defined particularly by shatter index and tumbler stability (table 23). From the modestly optimum coke quality produced by charging to the oven pre-heated to 100°C, both shatter index and tumbler stability decreased moderately with increase of charging temperature to a minimum in the vicinity of 300°C (fig. 62). In tumbler stability, a slight improvement in cokes formed from coals charged at 430°C was immediately followed by a decline which became precipitous for those charged above 540°C. The shatter index of cokes produced from coals charged at temperatures from 300°C to 540°C showed a quite marked improvement, but higher temperatures of charging were accompanied by greatly depreciated properties (fig. 62). Tumbler hardness and micromechanical strength indices, after some fluctuation, all improved appreciably and progressively in cokes produced from coals charged at temperatures higher than 650°C, a response to greater strength in the coke substance.

The greatly reduced shatter and tumbler stabilities of cokes produced from coals charged above 540°C is closely associated with the more extensive jointing and cross-fracturing developed in them. As revealed by the initial, intermediate, and final indices for shatter and tumbler stability, the breakdown of the coke increases greatly with the higher temperature of charging (table 23, figs. 63 and 64). In appearance, the cokes resulting from coals charged at the increasing temperatures exhibit progressive and marked differences in size characteristics and luster (pl. 4). Proximate analysis of the various cokes indicates a generally progressive increase of fixed carbon with increase of charging temperature (table 24).

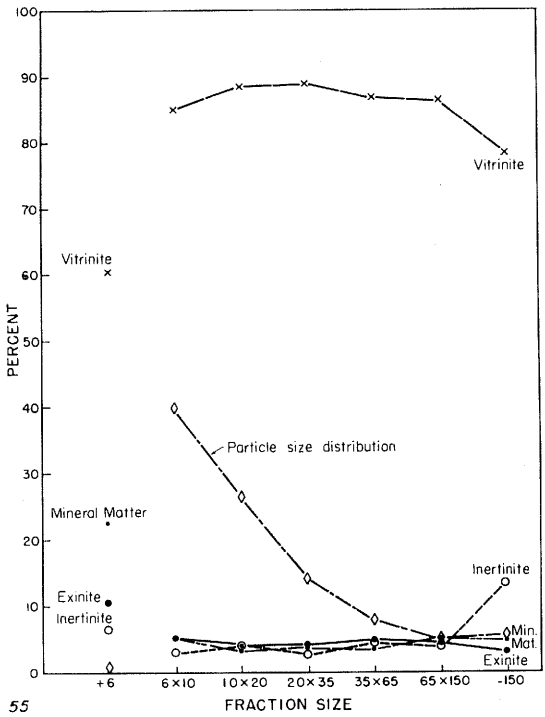
As in No. 6 coal, the optimum values of quality as determined by the various methods were not developed in any single coke and could not be related to a single charging temperature. On the basis of these results a compromise standard charging temperature of 540°C was adopted for all samples of the No. 5 coal.

FIG. 51.—No. 5 coal: Petrographic and chemical variation in reference pillar and seam sections (tables 20A and 21).

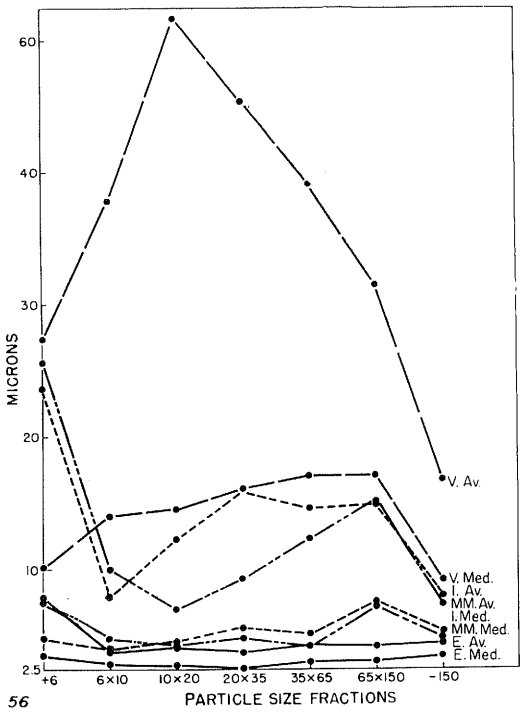
FIG. 52.—No. 5 coal: Fluidity values and swelling indices for reference pillar, seam sections I and II; also fluidity and swelling indices obtained from modified procedure (table 21). *See p. 45.

FIG. 53.—No. 5 coal: Petrographic constitution and chemical composition of individual size fractions of reference pillar. Standard preparation (tables 20B and 22).

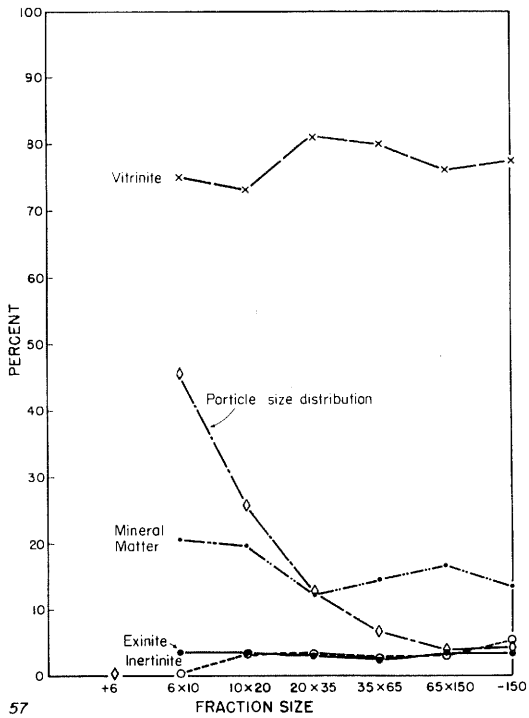
FIG. 54.—No. 5 coal: Fluidity values and swelling indices for size fractions of reference pillar; also fluidity and swelling indices obtained for modified procedure (table 22). *See p. 45.


PERCENT
Vitrinite
Particle size distribution
Mineral Matter
Inertinite
Exinite
Min. Mat.
+6
6×10
10×20
20×35
35×65
65×150
-150
FRACTION SIZE

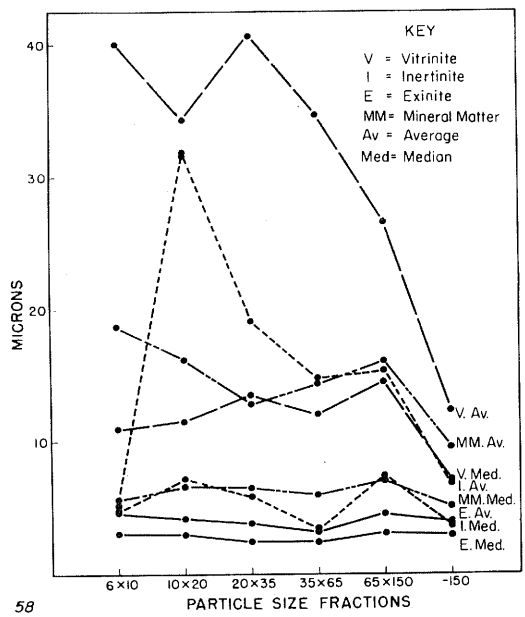
55


MICRONS
V. Av.
V. Med.
I. Av.
MM. Av.
I. Med.
MM. Med.
E. Av.
E. Med.
+6
6×10
10×20
20×35
35×65
65×150
-150
PARTICLE SIZE FRACTIONS

56


PERCENT
Vitrinite
Particle size distribution
Mineral Matter
Exinite
Inertinite
+6
6×10
10×20
20×35
35×65
65×150
-150
FRACTION SIZE

57


KEY
V = Vitrinite
I = Inertinite
E = Exinite
MM= Mineral Matter
Av = Average
Med= Median
MICRONS
V. Av.
MM. Av.
V. Med.
I. Av.
MM. Med.
E. Av.
I. Med.
E. Med.
6×10
10×20
20×35
35×65
65×150
-150
PARTICLE SIZE FRACTIONS

58

FIG. 55.—No. 5 coal: Maceral proportion variation in size fractions of reference pillar, standard preparation (tables 19, 20B).

FIG. 56.—No. 5 coal: Maceral size variation in size fraction of reference pillar, standard preparation (table 20B).

FIG. 57.—No. 5 coal: Maceral proportion variation in size fractions of seam section I, standard preparation (tables 19, 20B).

FIG. 58.—No. 5 coal: Maceral size variation in size fractions of seam section I, standard preparation (table 20B).

During these studies, however, a number of low anomalous values for shatter and tumbler indices were obtained from samples which developed an "extrusion plug" structure in the course of coking (pl. 6). In this feature an inner cylinder of well formed coke with open texture, approximately two inches in diameter, had moved along a cylindrical, highly polished glide plane, which marked the separation of the core from the co-axial outer cylinder, approximately one-half inch thick, of well formed denser coke. It is considered that these structures resulted from the formation of a gas seal in the plastic zone or "coking front" during the alteration from coal to coke. Gas evolution in the core of the incipiently altering coal charge is believed to have induced sufficient stress to cause rupture and movement along a glide plane

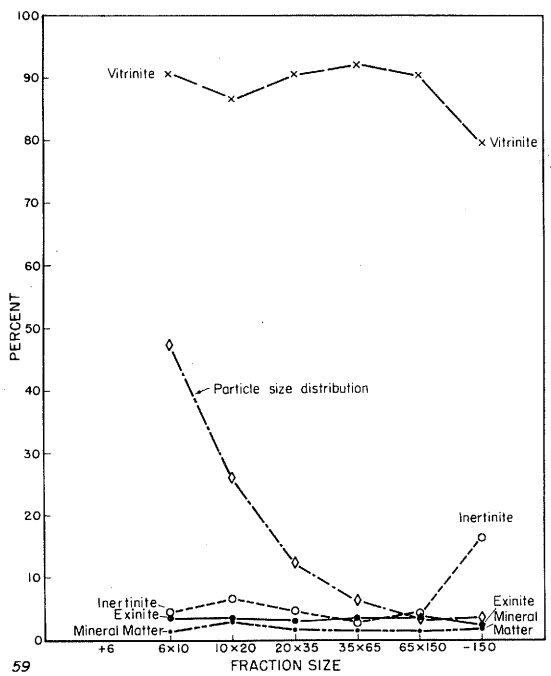
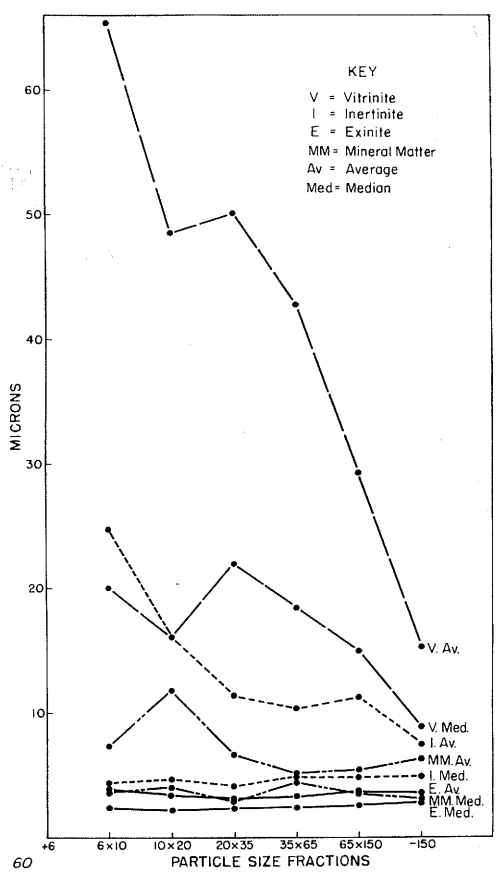


FIG. 59.—No. 5 coal: Maceral proportion variation in size fractions of seam section II, standard preparation (tables 19, 20B).

FIG. 60.—No. 5 coal: Maceral size variation in size fractions of seam section II, standard preparation (table 20B).



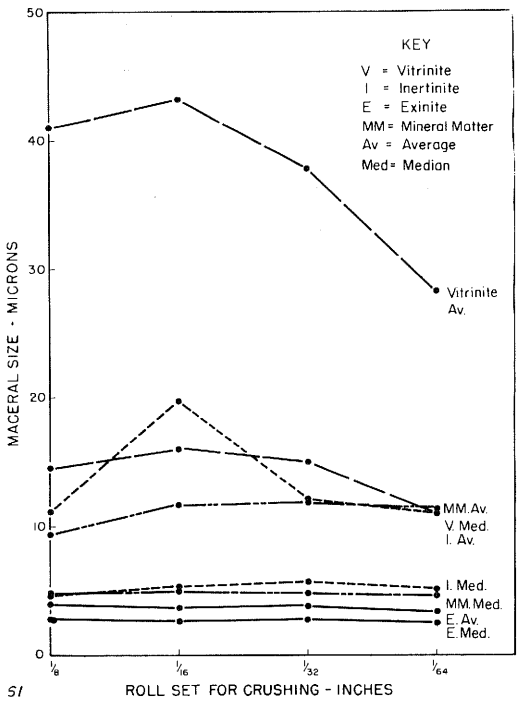


FIG. 61.—No. 5 coal: Progressive degradation of petrographic constituents (macerals and mineral matter) with finer degree of breaking, as expressed by mean and median maceral sizes. Reference pillar samples (table 20A).

developed in the plastic zone. As these structures developed principally in cokes formed from samples charged at the higher temperatures, in which a "gas seal" in the plastic zone would be most likely to form due to the more rapid rate of heating, it is now considered that a rather lower standard temperature of charging could with advantage have been selected for the subsequent test series. As in the No. 6 coal, the charging temperature in relation to the plastic range appears to have been significant, hence 450°C might have been a more suitable charging temperature.

Effect of Coking Temperature.—In this exploratory series, all samples were charged at the standard temperature of 540°C, raised at the normal rate of temperature increase (3.6°C per minute) to the final coking temperature selected, and maintained thereat for two hours. Standard samples of the representative pillar and bed sections I and II were each coked at final temperatures of 930°C, 1010°C and 1090°C (table 25, figs. 65, 66, 67). Under these different conditions of final coking temperature, the reference pillar samples produced cokes of varied character (table 25, fig. 65).

As indicated by shatter index and tumbler stability, those coked finally at 1010°C were definitely stronger, corresponding to a lesser development of jointing and fracture. Micro-mechanical strength indices and tumbler hardness, with one exception in the micro-strength 65 index, increased with the higher temperature of final coking, indicating the development of a stronger, tougher coke substance.

Samples of seam section I demonstrated "soaking" temperature-strength trends similar to the representative pillar with the exception of one value in the tumbler hardness index (table 25, fig. 66). Most notable, however, were the generally high values of the various indices of the high-ash cokes. It appears possible that when present in suitable proportions, form, and distribution, the mineral matter may act like inertinite and impart increased mechanical strength to the coke.

In contrast with those of the reference pillar and lower seam section, the cokes of the upper seam section (SS II) displayed a rather different relationship between macroscopic strength and temperature of final coking (table 25, fig. 67). Both shatter and tumbler stability indices were notably low in the highest coking temperature, possibly a

TABLE 25.—No. 5 COAL: INFLUENCE OF FINAL COKING TEMPERATURES ON COKE PRODUCED FROM REFERENCE PILLAR AND SEAM SECTIONS

(Standard size consist, charging temperature, rate of temperature increase, and final coking period)

Seam section and coking temp.			Macro-test data of cokes			Micro-test data of cokes	
				Tumbler		Mechanical strength	
	°C±	°F	Shatter: % + 1″	Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Reference pillar							
Coking temp:	930°	1700°	77.5	62.0	83.7	46.0	0.039
	930°	*	50.9				
	1010°	1850°	91.8				
	1010°	*	61.6	68.1	85.0	44.9	0.045
	1010°	†	81.2	70.3	84.6	40.9	0.044
	1090°	2000°	78.0	53.9	85.8	50.1	0.051
Seam section I							
Coking temp:	930°	1700°	89.7	88.9	88.9	44.6	0.121
	1010°	1850°	97.3	89.7	89.7	44.6	0.124
	1090°	2000°	89.5	79.5	79.7	51.2	0.144
Seam section II							
Coking temp:	930°	1700°	88.0	73.0	84.2	40.3	0.015
	930°	*	45.6	—	—	—	—
	1010°	1850°	71.7	62.8	84.5	41.9	0.019
	1090°	2000°	62.6	41.2	86.2	45.9	0.022
	1090°	*	29.4	—	—	—	—

*Extrusion plug—see plate 6.
†Mean standard. Temp. selected as standard for No. 5 coal, results are mean of several runs.

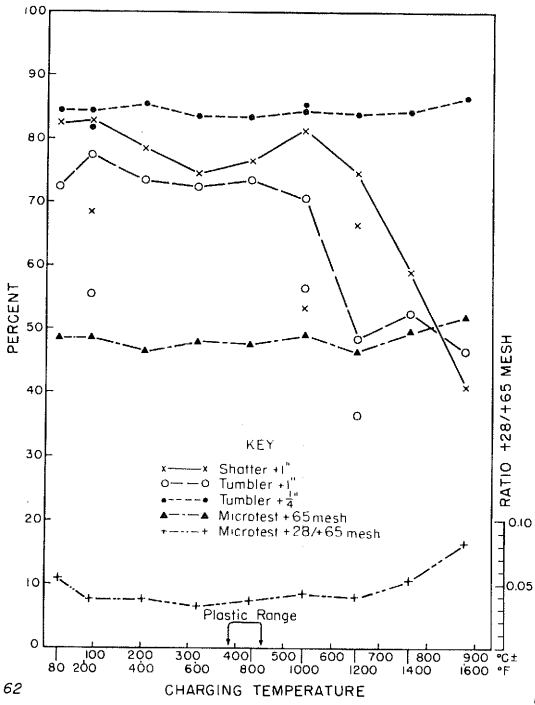
reaction to the greater stresses accompanying the volume changes associated with the coking of the considerable proportions of vitrinite in this coal. Although containing the highest proportion of inertinite, this may have been inadequate to compensate for the great volume changes brought about during the coking of the vitrinite, particularly at higher "soaking" temperatures.

It is significant that, in general, cokes of greater macro-strength (shatter and tumbler stability) were produced from samples of the reference pillar and lower bed section I in which, although the content of inertinite was less, finely divided disseminated mineral matter was present in greater proportions. In these two seam samples (RP and SS I), the combined proportions of inertinite and mineral matter exceed those of the upper seam section.

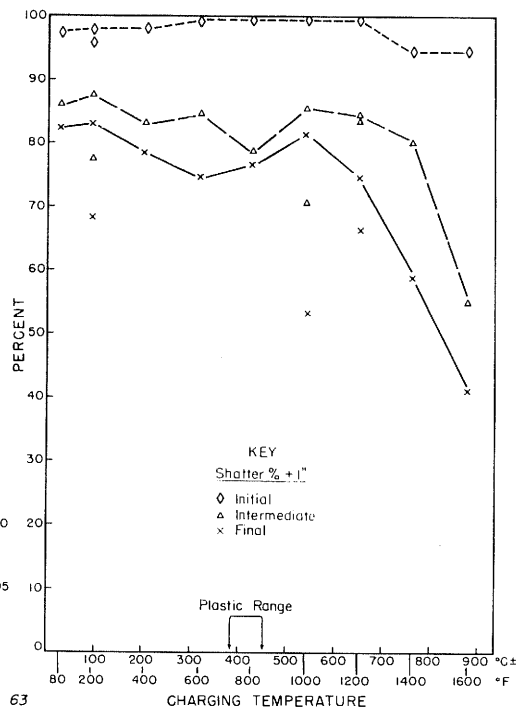
The micro-mechanical indices and tumbler hardness increased with temperature of final coking, demonstrating the development of a stronger structure in the actual substance of the coke.

Having due regard for all the circumstances of the present test series and the results thereof, 1010°C was accepted as the most appropriate final coking temperature for all further studies on the No. 5 coal, the same temperature that was used for No. 6 coal.

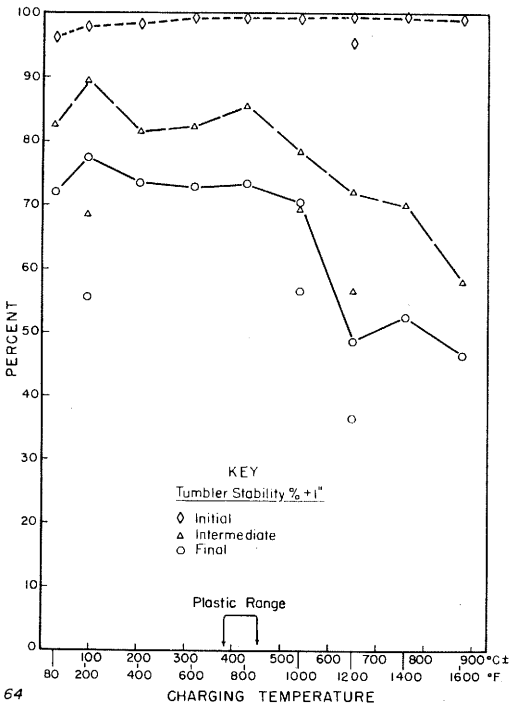
Of some interest are the fixed carbon, ash and sulfur variations exhibited by cokes from these seam sections as related to temperatures of final coking



62



63



64

FIG. 62.—No. 5 coal: Influence of charging temperature on coke. Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period 2 hours (table 23).

FIG. 63.—No. 5 coal: Influence of charging temperature on coke: progressive degradation in shatter test. Reference pillar, standard size consist, rate of temperature increase, final coking temperature and final coking period 2 hours (table 23).

FIG. 64.—No. 5 coal: Influence of charging temperature on coke: progressive degradation in tumbler test. Reference pillar, standard size consist, rate of temperature increase, final coking temperature, and final coking period 2 hours (table 23).

(table 26, fig. 68). Cokes of the upper and cleaner seam section (SS II) display the greatest increase of fixed carbon with coking temperature. Although showing a similar relationship to seam section I the fixed carbon of the reference pillar cokes increased more between 930°C and 1010°C, whereas that of the lower (dirty) seam section I cokes increased more between 1010°C and 1090°C. The sulfur-ash relationships were quite regular. Attempts to correlate fixed carbon content with coke strength were not particularly successful.

Effect of Final Coking Period.— Charged at temperatures of 540°C and brought by the standard rate of temperature increase to the final coking temperature of 1010°C, standard samples of the reference pillar were allowed to continue "soaking" for periods of one to six hours. In respect to shatter index, tumbler stability and hardness, those cokes which "soaked" for two hours proved to have the greatest strength (table 27, fig. 69). At longer periods, the tumbler stability and hardness decreased progressively; the shatter index appeared erratic. The shorter period of one hour produced cokes of inferior quality on all three bases of assessment.

As determined by the results of these three study series, the standard procedures adopted for all subsequent investigations concerned with the production of coke from samples of the No. 5 coal were defined as:

Charging temperature	540°C.
Rate of temperature increase	3.6°C/min.
Final coking temperature	1010°C
Final coking period	2 hours

These standard conditions were the same as for the No. 6 coal except the temperature of charging (450°C for No. 6 coal).

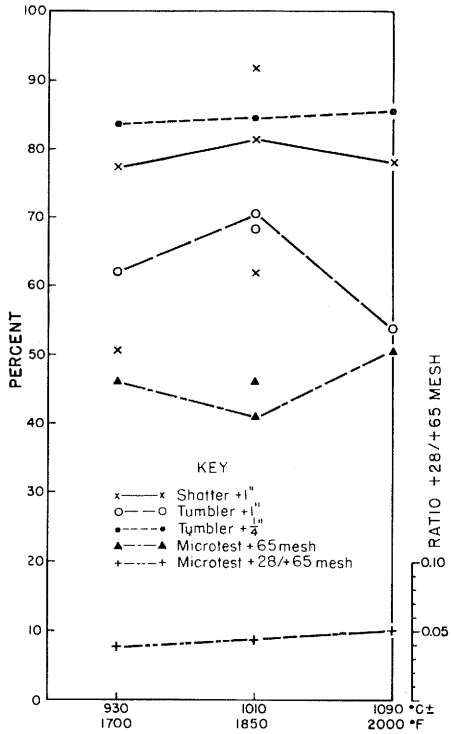
Influence of Petrographic Constitution

In the absence of macro-type samples, attempts to correlate coke characteristics with the petrographic character of the original coal sample from which it was produced had to be confined to the seam sections (RP, SS I and SS II) (tables 20A, fig. 70). The increases in shatter index, tumbler stability and hardness, as well as of the micro-mechanical strength indices, with one exception, can be related to decrease in the vitrinite content and increase in the proportions of the total "inerts" (inertinite and mineral matter combined); the proportion of inertinite, however, to total "inerts" was less in the pillar sample and seam section I from which the strongest cokes were prepared. In all probability the individual inertinite proportions were too small to produce appreciable modification in the cokes of these high vitrain coals.

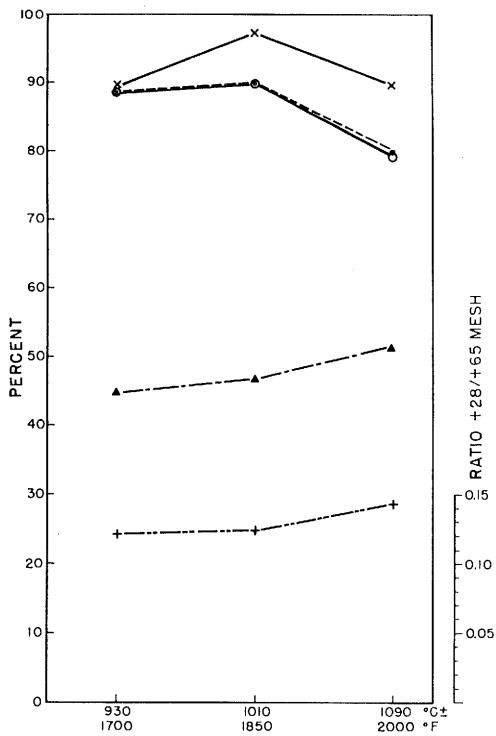
With so few samples to consider, efforts to relate the mechanical properties of the cokes to both constituent proportions and size characteristics as determined by median and quartile values were ineffective.

Influence of Coal Particle Size Fractions

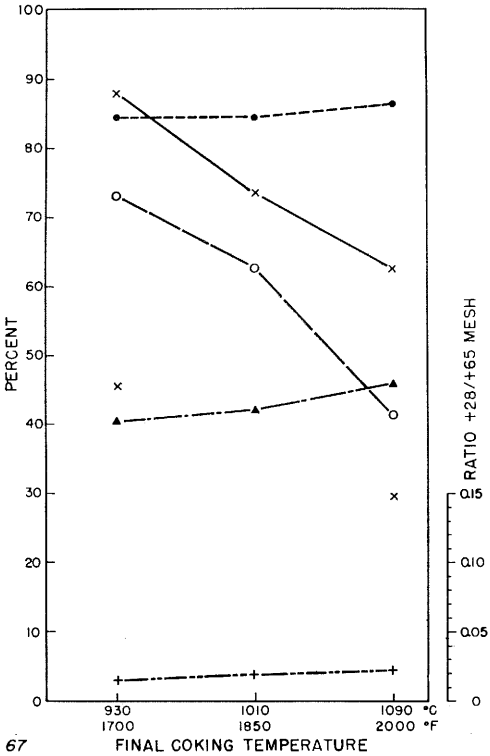
As in the No. 6 coal, the petrographic analyses of the seam samples of No. 5 coal revealed that differential breakage in the course of preparation produced some selective variation in the proportions of the macerals present in the different size fractions of each standard sample (table 20B, figs. 53 and 55). In general, there is a pronounced tendency for the coarser material of the broken


PERCENT
RATIO +28/+65 MESH
KEY
Shatter +1"
Tumbler +1"
Tumbler +¼"
Microtest +65 mesh
Microtest +28/+65 mesh
930 1700
1010 1850
1090 2000
°C±
°F
FINAL COKING TEMPERATURE

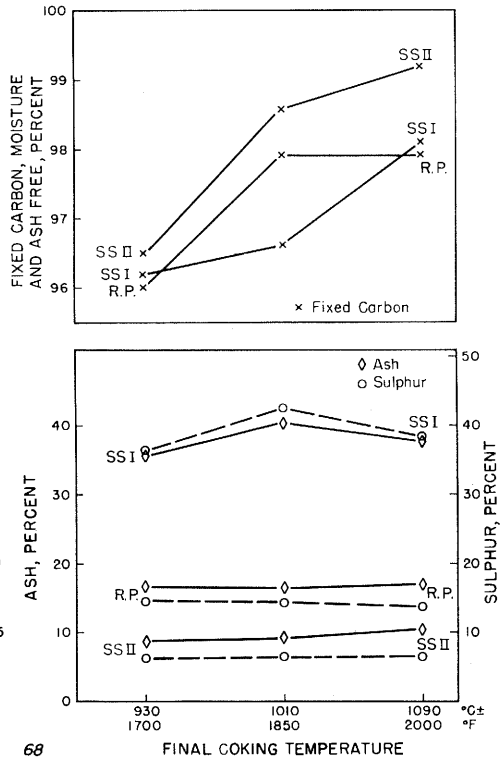
65


PERCENT
RATIO +28/+65 MESH
930 1700
1010 1850
1090 2000
°C±
°F
FINAL COKING TEMPERATURE

66


PERCENT
RATIO +28/+65 MESH
930 1700
1010 1850
1090 2000
°C
°F
FINAL COKING TEMPERATURE

67


FIXED CARBON, MOISTURE AND ASH FREE, PERCENT
SS II
SS I
R.P.
× Fixed Carbon
◊ Ash
o Sulphur
ASH, PERCENT
SULPHUR, PERCENT
930 1700
1010 1850
1090 2000
°C±
°F
FINAL COKING TEMPERATURE

68

coal to be formed from the more finely textured coal types. To the extent that this is true the coking results must be influenced by both size characteristics and petrographic composition of the coal charges.

The variations in the mechanical properties of the cokes produced from individual size fractions of the standard reference pillar sample were impressive (table 28, fig. 71, pl. 5, A to F). From the relatively low values for the coke produced from the plus 6-mesh fraction, the shatter index and tumbler stability increased notably to a maximum in the 35 x 65 mesh range and then declined markedly to the minus 150-mesh fraction. The tumbler hardness followed the same trend generally but less emphatically. The micro-strength 65 index decreased progressively with diminishing particle size over the entire size range, but most strongly in the 65 x 150 mesh fraction; the ratio 28/65 of the micro-mechanical tests remained reasonably constant in plus 20-mesh material but thereafter decreased markedly.

Exploration of the possibility that petrographic or chemical variations among the samples might be at least partly responsible for the variations in the mechanical properties of the cokes was made by plotting pertinent data in relation to coke properties in the graphs shown in figures 72, 73, and 74 (tables 20B, 22, and 28) respectively. Although there is possibly a broad and imperfect correspondence in the trend of variation between vitrinite content and coke macro-strength indices, or antithetical relation between these characteristics and the "inerts," the individual relationship of the specific characteristics is too ill-defined to be satisfactory (tables 20B, 28, fig. 72). A study of the proportions of coking and non-coking coal constituents compared on a group basis with coke properties was likewise unsatisfactory.

Attempts to relate chemical characteristics, fluidity values (Gieseler and modified test) and free swelling indices (standard test and modified test) with the mechanical properties of the cokes were similarly unrewarding (tables 20B, 22, 28; figs. 73 and 74).

It thus appears that properties of the cokes produced from the individual coal size fractions must be mainly a function of the coal particle size, as was believed to be the case in the similar test series completed on the No. 6 coal. The greatly increased porosity of the samples of the finer size coal fractions would be a factor accounting for the great decrease of strength (macro and micro) in the cokes produced from size fractions below 65-mesh. The low macro-strength characteristics of the coarse fractions

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FIG. 65.—No. 5 coal: Influence of final coking temperature on coke. Reference pillar, standard size consist, charging temperature, rate of temperature increase, and final coking period 2 hours (table 25).

FIG. 66.—No. 5 coal: Influence of final coking temperature on coke. Seam section I, standard size consist, charging temperature, rate of temperature increase, and final coking period 2 hours (table 25).

FIG. 67.—No. 5 coal: Influence of final coking temperature on coke. Seam section II, standard size consist, charging temperature, rate of temperature increase, and final coking period 2 hours (table 25).

FIG. 68.—No. 5 coal: Fixed carbon, ash, and sulphur in cokes produced from reference pillar and seam sections at various temperatures of final coking (table 26).

TABLE 26.—No. 5 COAL: PROXIMATE ANALYSES OF COKE PRODUCED BY VARIOUS FINAL COKING TEMPERATURES
(See table 25)

Sample	Final coking temp.		Air dried							Moisture and ash free			
	°C±	°F	Coke yield %*	Moist.	Ash	Vol.	F. C.	S	Btu/lb.	Vol.	F. C.	S	Btu/lb.
Reference pillar	930	1700	72	3.5	16.5	3.2	76.8	1.18	11386	4.0	96.0	1.48	14233
Seam section I	930	1700	74	3.2	35.6	2.3	58.9	2.22	—	3.8	96.2	3.63	—
Seam section II	930	1700	72	3.7	8.8	3.1	84.4	0.53	—	3.5	96.5	0.61	—
Reference pillar	1010	1850	69	0.9	16.5	1.7	80.9	1.18	—	2.1	97.9	1.43	—
Seam section I	1010	1850	72†	1.4	40.4	2.0	56.2	2.47	—	3.4	96.6	4.24	—
Seam section II	1010	1850	70	0.9	9.1	1.3	88.7	0.57	—	1.4	98.6	0.63	—
Reference pillar	1090	2000	71	0.6	16.9	1.7	80.8	1.15	—	2.1	97.9	1.39	—
Seam section I	1090	2000	73	0.5	37.7	1.2	60.6	2.35	—	1.9	98.1	3.80	—
Seam section II	1090	2000	72	0.9	10.5	0.7	87.9	0.57	—	0.8	99.2	0.64	—

*Average of three test samples, not adjusted for moisture.
†Average of two test samples, not adjusted for moisture.

TABLE 27.—No. 5 COAL: INFLUENCE OF PERIOD OF FINAL COKING ON COKE
(Reference pillar, standard size consist, charging temperature, rate of temperature increase, and final coking temperature)

Period of final coking at 1010°C–1850°F (Hours)	Macro-test data of cokes			Micro-test data of cokes	
	Shatter: % + 1″	Tumbler		Mechanical strength	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
1.	70.2	65.9	79.6	‡	
1.*	57.5	—	—		
2.†	81.2	70.3	84.6	46.9	0.044
2.*	53.1	56.2	85.4		
4.	72.9	67.1	83.7	‡	
6.	78.6	56.7	82.6	‡	

*Extrusion plug—see plate 6.
†Mean standard. Selected as standard for No. 5 coal, results are mean of several tests.
‡Micro tests were not made on these cokes.

TABLE 28.—No. 5 COAL: INFLUENCE OF INDIVIDUAL AND CUMULATIVE SIZE FRACTIONS ON COKE
(Reference pillar, standard coking conditions)

Coal size fraction	Macro-test data of coke			Micro-test data of coke	
	Shatter: % + 1″	Tumbler		Mechanical strength	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
+6 mesh	35.8	20.6	73.5	49.1	0.040
6 x 10	51.4	49.5	80.7	47.1	0.038
10 x 20	75.8	59.0	79.4	46.2	0.043
20 x 35	84.2	67.6	82.6	42.7	0.017
35 x 65	90.7	74.1	85.9	37.3	0.005
65 x 150	84.9	34.7	65.2	6.9	0
−150	15.8	9.7	34.9	0.1	0
Cumulative size fraction					
All +20	74.6	65.2	80.7	46.9	0.039
All +35	60.8	56.7	81.1	47.6	0.035
All +65	66.8	51.0	84.4	44.9	0.040
All +150	75.1	74.9	84.4	44.6	0.042
Entire sample	87.7	72.1	85.4	46.3	0.039

TABLE 29.—No. 5 COAL: SIZE DISTRIBUTION, INDIVIDUAL AND CUMULATIVE, RESULTING FROM SELECTIVE AND STANDARD BREAKAGE
(Reference pillar, proportions in percentage)

Sample	(a)		(b)		(c)		(d)		(e)		(f)*	
Ratio $\frac{+65}{-65}$	3.0		3.9		4.2		5.3		6.7		8.3	
Size range	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.	Ind.	Cum.
+10 mesh	0.51	0.51	20.6	20.6	2.30	2.30	40.4	40.4	15.38	15.38	40.6	40.6
10 x 20	12.71	13.22	14.5	35.1	31.86	34.16	4.4	44.8	42.22	57.60	26.5	67.1
20 x 35	38.53	51.75	24.2	59.3	30.65	64.81	22.8	67.6	19.31	76.91	14.1	81.2
35 x 65	23.24	74.99	20.3	79.6	15.99	80.80	16.5	84.1	10.17	87.08	8.0	89.2
65 x 150	12.31	87.30	10.0	89.6	9.46	90.26	7.8	91.9	6.17	93.25	5.0	94.2
−150	12.70	100.00	10.4	100.0	9.74	100.00	8.1	100.0	6.75	100.00	5.8	100.0

*Standard size consist.

TABLE 30.—No. 5 COAL: INFLUENCE OF SELECTIVE AND STANDARD BREAKAGE ON COKE
(Reference pillar, standard coking conditions)

Coal size "factor" Ratio $\frac{+65}{-65}$	Macro-test data of coke			Micro-test data of coke	
	Shatter: % + 1″	Tumbler		Mechanical strength	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
(a) 3.0	68.9	53.8	86.3	47.0	0.033
(b) 3.9	77.2	60.5	85.4	47.1	0.030
3.9*	53.8	—	—	—	—
(c) 4.2	85.6	50.2	84.9	45.8	0.039
(d) 5.3	77.0	71.4	85.3	44.4	0.029
(e) 6.7	69.7	69.1	85.9	47.1	0.052
6.7*	61.3	—	—	—	—
(f) 8.3†	81.2	70.3	84.6	46.9	0.044
8.3*	53.1	56.2	85.4	—	—

*Extrusion plug.
†Mean standard.

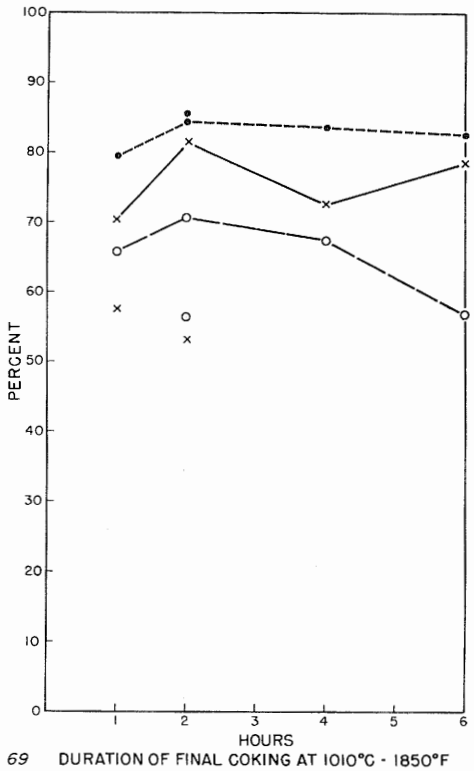
most probably reflect a severe decrease of areas of contact where fusion can take place. The generally high level of the micro-strength of cokes formed from these coarser fractions was largely determined by the circumstances of the test whereby small coke masses were formed from individual coal particles.

The initially constant and more gradual decline in the respective micro-strength indices with decrease in particle size (table 28, fig. 71) may be related to the petrographic constitution and maceral size distribution in the various sizes. In the broken coal, vitrinite proportions and median sizes increase with decrease in particle size; inertinite and mineral matter exhibit a similar trend. It is evident that the coarser size fractions of the broken coal are produced preferentially from the finer banded, finer "grained" coal types. In the finer coal types there is generally a better, more uniform distribution of the macerals and in consequence they are more intimately related. This generally better distribution and closer association of coking and "inert" constituents in the coarser fractions is considered to have contributed to the greater micro-strength of the coke substance. The increase in median size of the macerals in the finer fractions would be associated with their less effective distribution, less intimate relationship and consequently with the production of a coke substance of inferior micro-mechanical properties.

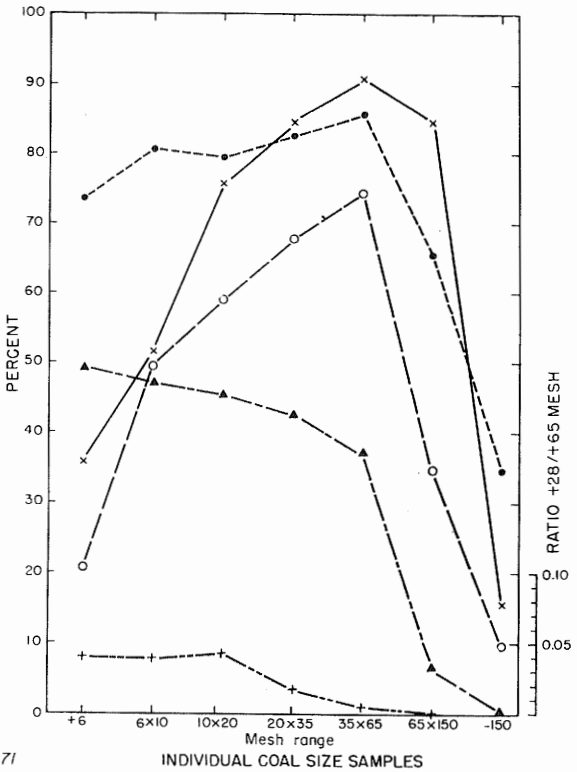
Coke produced from cumulative size fractions (table 28, fig. 75), although demonstrating the importance of the finer fractions to the production of better coke (improved "bonding"), introduced an apparently systematic variation which did not conform to the normal trend of noncumulative shatter index and tumbler stability (table 20B, fig. 72) which was reversed until the minus 65- and minus 150-mesh material was included in the charge.

Influence of Coal Size Consist

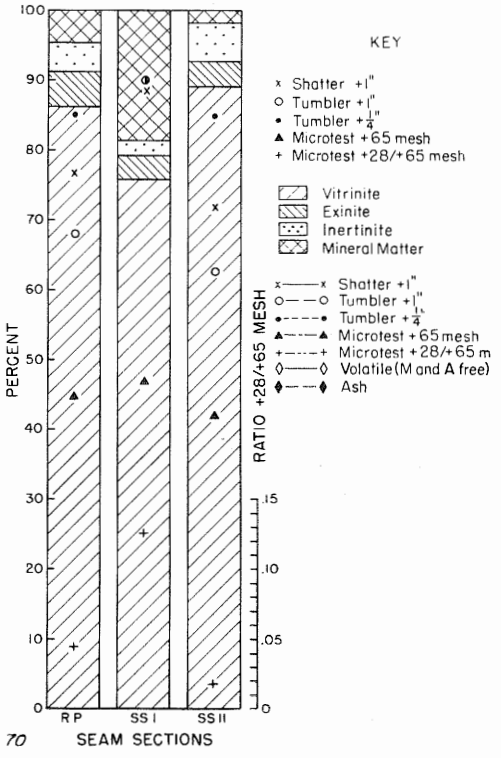
The effects of variation in coal size consist in the retort charge upon the character of the coke produced, proved to be of some importance in the case of the No. 6 coal, but were not necessarily in strict accordance with the pre-



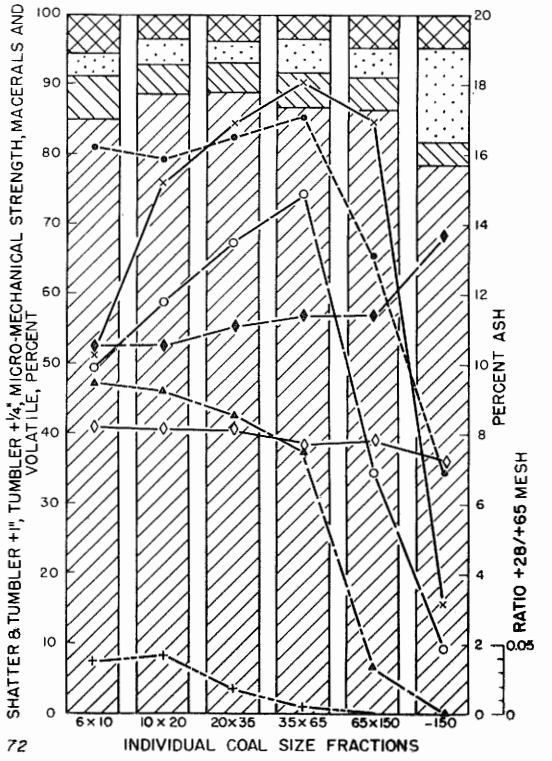
69 DURATION OF FINAL COKING AT 1010°C - 1850°F



71 INDIVIDUAL COAL SIZE SAMPLES



70 SEAM SECTIONS



72 INDIVIDUAL COAL SIZE FRACTIONS

ferred consist determined by Burstlein for the coals of the Saar and Lorraine.

Exploration of the coal size consist factor in relation to the properties of coke produced from the No. 5 coal was limited to a series of six specially prepared samples of which the characteristics and cumulative size curves appear in table 29 and figure 76, respectively. Characteristics of the cokes produced from these charges under standard conditions of coking are summarized in table 30 and figure 77 (pl. 5, G to L). The size distribution within the coal is rather inadequately represented by the +65/–65 mesh ratio; yet this size appears from previous studies to be of value in the determination of coke quality.

With decrease in the proportion of fines in the retort charge, the macrostrength properties generally improve. The apparently anomalous values for the tumbler stability of cokes produced from charges (b) and (c) may be related to the difference in the relative proportions of the larger material represented mainly by the plus 10-mesh fraction. The dominance of the larger size material in (b) would favor formation of a more resistant coke substance which would influence the stability of the coke. In support of this explanation it is noteworthy that the micromechanical indices are also slightly increased in (b) as compared with (c). The reduced shatter indices for the cokes (d) and (e) can possibly be related to the lack of optimum proportions of coarse and intermediate size fractions which is evident in the original coal charges.

As regards over-all mechanical characteristics of the cokes, the coal size distribution produced by the controlled and limited breakage adopted within a given size range of plus 6- to minus 150-mesh appeared to be the most satisfactory. The size consist of the coal samples prepared by the standard initial procedure yielded cokes in which the various strength factors were most satisfactorily combined ("f" on table 29, fig. 77, and pl. 5, J).

Influence of Fusain

In the absence of macro-type samples of the No. 5 coal, additional fusain of minus 150-mesh was blended in proportions of up to 25 percent with standard (minus ⅛ inch) and reduced size (minus 1/16 inch) consist, samples of both reference pillar and upper seam section (SS II). By examining the effects of fusain blended with identical samples of coal crushed to different size consists, it was hoped to obtain information upon the possibility of utilizing "fines" high in fusain for the production of coke.

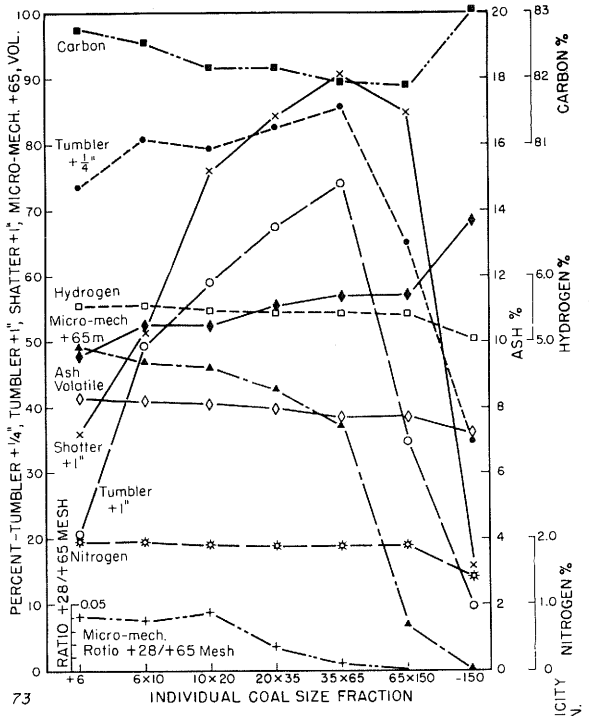
The fusain used for blending was obtained from a number of conspicuous lenses up to 2 inches thick and several

FIG. 69.—No. 5 coal: Influence of final coking period on coke. Reference pillar, standard size consist, standard charging temperature, rate of temperature increase, and final coking temperature (table 27).

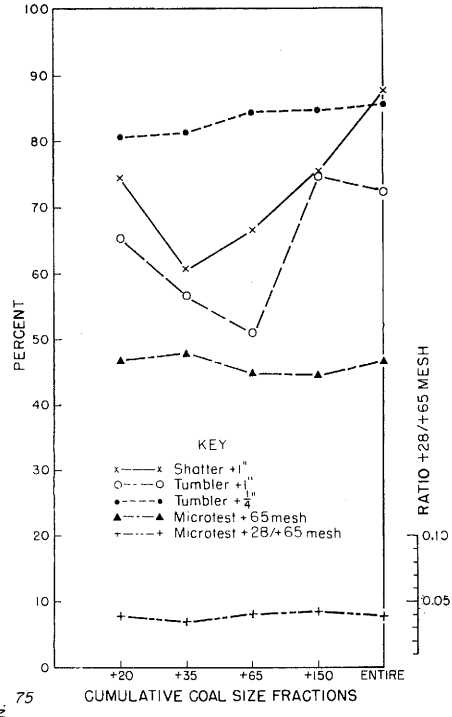
FIG. 70.—No. 5 coal: Influence of seam sections on coke. Reference pillar, seam section I, seam section II, standard size consist, standard coking conditions (tables 20A and 25).

FIG. 71.—No. 5 coal: Character of coke produced from individual size fractions. Reference pillar, standard coking conditions (table 28).

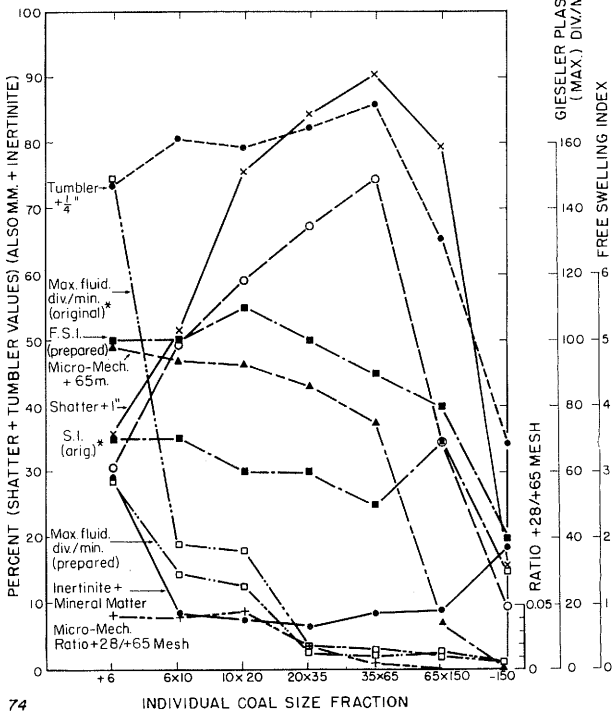
FIG. 72.—No. 5 coal: Character of coke produced from individual size fractions, related to petrographic constitution, volatile matter and ash. Reference pillar, standard coking conditions (tables 20B, 22, and 28).



73



75



74

FIG. 73.—No. 5 coal: Character of coke produced from individual size fractions, related to chemical constitution. Reference pillar, standard coking condition (tables 22, 28).

FIG. 74.—No. 5 coal: Character of cokes and amount of inertinite plus mineral matter produced from individual size fractions related to Gieseler fluidity and free swelling indices; also fluidity and swelling indices obtained from modified procedures. Standard coking conditions (tables 22 and 28). *See p. 45.

FIG. 75.—No. 5 coal: Character of coke produced from cumulative size fractions. Reference pillar, standard coking conditions (table 28).

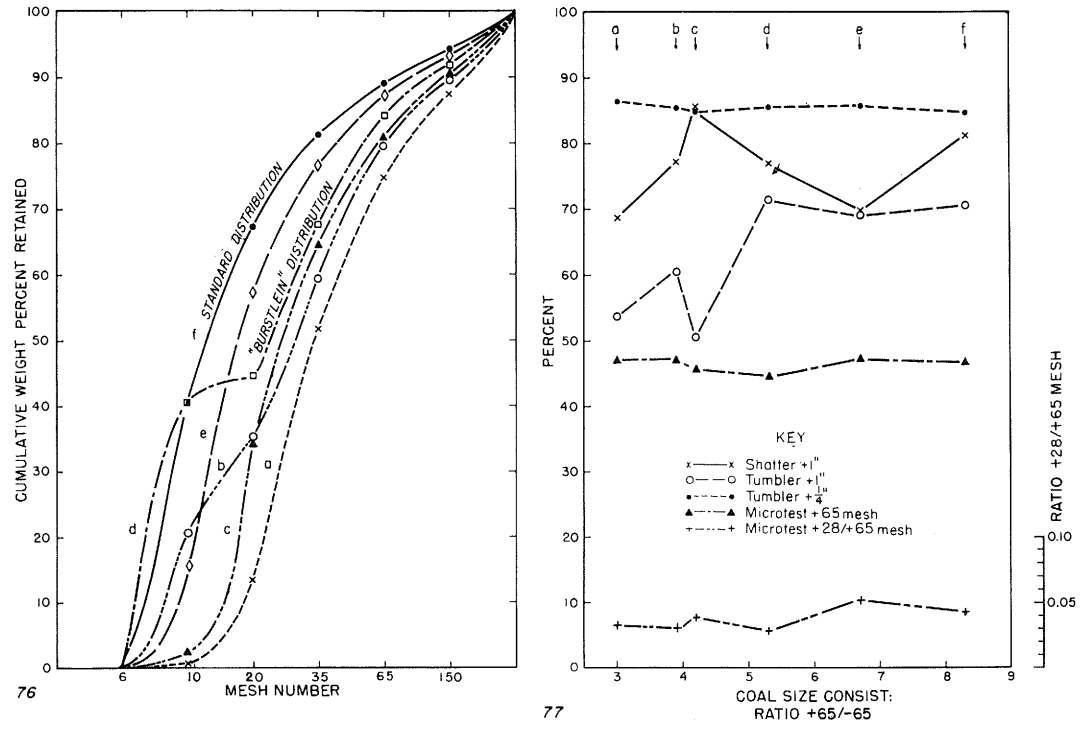


FIG. 76.—No. 5 coal: Influence of selective breakage on distribution of particle size. Reference pillar, size consist of individual charges coked. Standard coking conditions. Use with Fig. 77 (table 29).

FIG. 77.—No. 5 coal: Influence of size consist on coke. Reference pillar, standard coking conditions. Use with Fig. 76 (table 30).

square feet in area, occurring in the vicinity of the site from which the reference pillar was extracted. Each lens represented an aggregate of fusain fragments and chips with very minor proportions of vitrain in thin irregular sheets. Immediately upon extraction the material was placed in air-tight cans until required for preparation.

Fusain Sample Preparation.—After hand picking to remove all possible vitrain, mineral matter, and other adventitious materials, the bulk fusain was passed once through the crusher with the rolls set at 1/16th inch separation. The material was then dried and screened, and only the minus 150-mesh fraction was used for blending. Micropetrographic analysis of this portion of the fusain is shown in figure 46A (table 20A).

In each blended sample, care was taken to insure uniformity of distribution of the components throughout the charge.

Blends of Reference Pillar and Minus 150-Mesh Fusain: Blends with Samples of Standard and Reduced Size Consists. Cokes made from reference pillar samples of standard size consist blended with 5 percent of minus 150-mesh additional fusain showed a slight decline in both shatter index and tumbler stability; tumbler hardness and micro-mechanical strength indices increased slightly (table 31, fig. 78). Increase in

TABLE 31.—No. 5 COAL: INFLUENCE ON COKE OF FUSAIN BLENDED WITH SAMPLES OF STANDARD AND SUB-STANDARD SIZE CONSIST
(Reference pillar and seam section II. Standard coking conditions)

Sample, condition and blend		Macro-test data of coke			Micro-test data of coke	
			Tumbler		Mechanical strength	
Percent	Percent	Shatter: % + 1″	Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
Reference pillar Crushed at ⅛″, standard size consist	−150 Fusain					
100	0	79.3	66.2	83.5	47.2	0.053
95	5	78.2	63.5	84.2	47.8	0.055
95	5*	48.7	—	—	—	—
90	10	81.6	85.9	85.9	45.9	0.040
85	15	93.7	83.0	84.0	46.9	0.050
85	15*	86.6	—	—	—	—
80	20	91.9	74.9	75.2	40.8	0.047
75	25	83.8	52.9	54.5	40.0	0.034
75	25*	59.8	—	—	—	—
Reference pillar Recrushed at 1/16″	−150 Fusain					
100	0	85.7	78.3	83.8	46.2	0.055
95	5	88.4	74.9	86.6	45.9	0.042
95	5*	80.1	—	—	—	—
90	10	92.7	73.0	85.4	45.7	0.045
90	10*	78.7	—	—	—	—
85	15	93.8	85.8	86.6	45.7	0.051
80	20	89.4	85.8	86.2	45.8	0.073
75	25	94.0	80.0	80.0	45.8	0.070
Seam section II Crushed at ⅛″, standard size consist	−150 Fusain					
100	0	71.7	62.8	84.5	41.9	0.019
100	0*	51.8	43.0	84.7	—	—
95	5	84.6	—	—	45.9	0.029
95	5*	64.3	67.2	87.0	—	—
90	10	90.2	74.6	86.1	44.1	0.026
85	15	92.5	80.8	84.8	47.6	0.039
80	20	89.3	81.8	82.0	48.9	0.045
75	25	81.8	73.3	73.3	41.9	0.018
Seam section II Recrushed at 1/16″	−150 Fusain					
100	0	81.9	58.8	84.1	44.2	0.018
95	5	83.8	69.0	86.9	43.7	0.026
90	10	86.1	69.8	85.8	45.2	0.023
85	15	94.3	85.5	85.6	39.2	0.018
80	20	93.8	84.6	84.6	41.6	0.030
80	20*	85.8	—	—	—	—
75	25	92.0	65.3	74.1	38.4	0.025

*Extrusion plug—see plate 6.

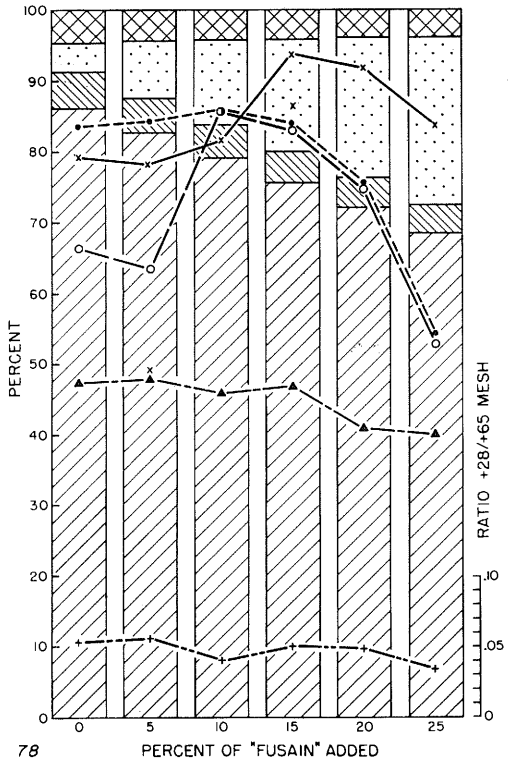
fusain proportions to 10 percent of the charge induced a considerable improvement in the maximum tumbler stability and hardness recorded for this coke series, with a much more modest gain in shatter index and a decline in micro-strength. At 15 percent fusain content, the shatter index reached a very satisfactory maximum with a slight decline in tumbler strength and slight improvements in micro-mechanical strength. Further increase in the fusain proportions was accompanied by a pronounced decline in appearance and all strength indices of the cokes in this test series (pl. 7, A to F).

When blended with standard samples of the reference pillar recrushed between rolls set at 1/16 inch, the fusain produced slightly different effects (table 31, fig. 79). The tumbler stability of the cokes decreased progressively in samples containing 5 percent and 10 percent of the minus 150-mesh fusain; it improved greatly with 15 percent and 20 percent to values comparable with the best of the previous series and much better than the cokes of the corresponding blends in that group. Cokes produced from charges containing 25 percent of the minus 150-mesh fusain declined in tumbler stability but not to the same degree as cokes made from the coarser sample of standard size consist. The shatter indices of the cokes produced from these fusain-coal blends of reduced size consist, with one exception, improved progressively with fusain content, increasing up to 25 percent of the charge; the cokes proved very resistant to disintegration under the test conditions (pl. 7, G to L).

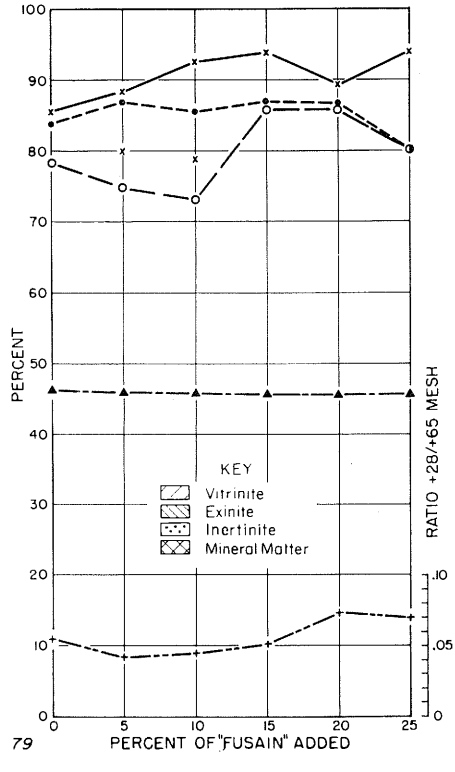
With fusain content equal to 5 percent of the charge, tumbler hardness of the coke improved, and with further increases up to 20 percent this quality was generally maintained. The micro-strength 65 index after a very slight initial decline remained almost constant throughout the series; the micro-strength factor represented by the 28/65 mesh ratio also decreased initially and thereafter increased to the highest values for cokes produced from blends with 20 percent and 25 percent additional fusain.

Blends of Seam Section II and Minus 150-Mesh Fusain: Blends with Samples of Standard and Reduced Size Consists. Petrographic examination had demonstrated an increased proportion of vitrinite in the standard samples of the upper seam section (SS II) as compared with those of the lower seam section (SS I) and with the reference pillar (RP) (table 20A). The possibility that cokes prepared from samples of seam section II, with an appreciably higher vitrinite content, when blended with fusain might exhibit significantly different properties (as in the case of the No. 6 coal) was amply substantiated (table 31, figs. 80, 81).

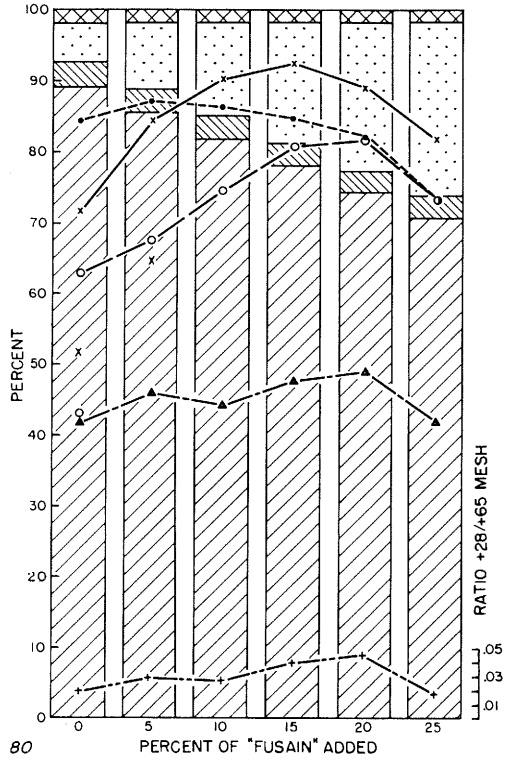
In respect to tumbler stability and micro-mechanical strength, blends of the upper seam section (SS II) of standard size consist (minus ⅛ inch) with increasing proportions of minus 150-mesh fusain up to 20 percent of the total charge produced cokes of substantially improved quality (fig. 80, pl. 8, A to F); further increase to 25 percent fusain resulted in decline of these coke indices. Tumbler hardness improved with 5 to 10 percent fusain in the charge but thereafter declined progressively as the proportions increased. Variation in the coke shatter index was particularly regular throughout the series reaching a maximum with 15 percent of additional fusain in the coal charge. The optimum values for shat-


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED

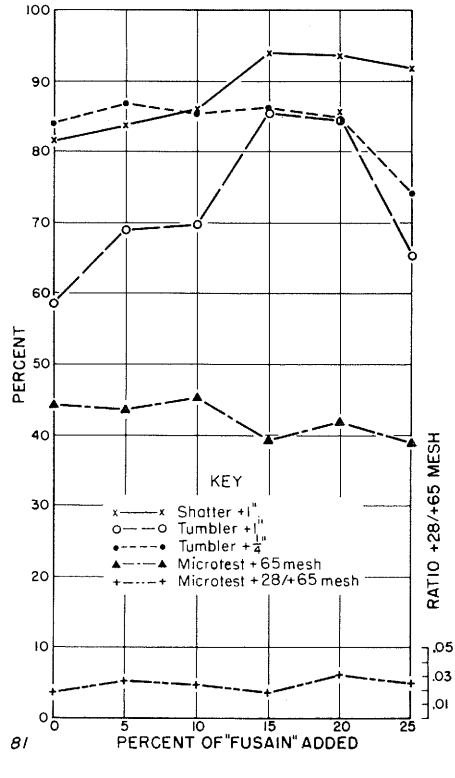
78


PERCENT
RATIO +28/+65 MESH
KEY
Vitrinite
Exinite
Inertinite
Mineral Matter
PERCENT OF "FUSAIN" ADDED

79


PERCENT
RATIO +28/+65 MESH
PERCENT OF "FUSAIN" ADDED

80


PERCENT
RATIO +28/+65 MESH
KEY
Shatter +1"
Tumbler +1"
Tumbler +¼"
Microtest +65 mesh
Microtest +28/+65 mesh
PERCENT OF "FUSAIN" ADDED

81

ter index and tumbler stability recorded in this series were not quite as good as for the fusain blends with reference pillar samples at either standard or reduced size consist, but the greatest tumbler hardness figures were about the same.

At reduced size consist, effected by recrushing the standard sample of the upper seam section at 1/16 inch roll separation, a corresponding, substantial, but less regular improvement in principal macro-strength characteristics of the cokes accompanied an increase in the additional fine fusain content up to 15 percent of the charge (fig. 81, pl. 8, G to L). Tumbler stability of the coke declined sharply with 25 percent fusain in the charge, but the shatter index fell only slightly. Tumbler hardness registered a maximum for coke produced from charges containing 5 percent additional fusain; with progressively higher contents of this maceral the index decreased slowly until at 25 percent fine fusain the quality of the resultant coke was substantially impaired. Although rather erratic, the micro-strength 65 index decreased generally with increase in the proportion of fine fusain, whereas the 28/65 ratio tended to improve.

As compared with the cokes produced from blends of fine fusain and upper seam section (SS II) samples of standard size consist (minus 1/8 inch), those of the present blend series (minus 1/16 inch) have proved to be as good as or even superior insofar as macro-strength is concerned; micro-strength was, however, appreciably less in the higher fusain range.

The effects of increased contents of fine fusain up to 15 percent in the retort charge appear to be generally beneficial insofar as macro-strength properties of the coke are concerned; exceptions occurred in the tumbler stability index for cokes produced from charges with up to 5 percent or 10 percent additional fusain. The effects were generally beneficial, in spite of the increase of "fines" produced by the higher additions of fusain.

With the notable exception of the cokes produced from blends with standard samples of the upper seam section, the micro-strength 65 index decreased generally with increase of fine fusain. On the other hand, with the exception of standard reference pillar-fusain blends, the micro-strength index 28/65 showed a general tendency to increase with proportions of additional fusain up to 20 percent; higher concentrations inevitably were accompanied by a decrease in this index.

The addition of fine fusain to the retort charges apparently contributes to the control of the changes of volume which take place during coking, and in propitious circumstances can add to the strength of the coke substance.

Within the limits of the present study, the reduced size consist of the standard samples seemed to be gener-

FIG. 78.—No. 5 coal: Influence of fusain on coke. Reference pillar, standard size consist, fusain minus 150-mesh, standard coking conditions (table 31).

FIG. 79.—No. 5 coal: Influence of fusain on coke. Reference pillar, reduced size consist (rebroken at 1/16 inch); fusain minus 150-mesh. Standard coking conditions (table 31).

FIG. 80.—No. 5 coal: Influence of fusain on coke. Seam section II, standard size consist; fusain minus 150-mesh. Standard coking conditions (table 31).

FIG. 81.—No. 5 coal: Influence of fusain on coke. Seam section II, reduced size consist (rebroken at 1/16 inch); fusain minus 150-mesh. Standard coking conditions (table 31).

TABLE 32.—No. 5 COAL: PROXIMATE ANALYSES OF COKE FROM BLENDS OF REFERENCE PILLAR AND MINUS 150-MESH FUSAIN

Sample proportions %		Air dried							Moisture and ash free			
Reference pillar	−150 mesh fusain	Coke yield %*	Moist.	Ash	Vol.	F. C.	S	Btu/lb.	Vol.	F. C.	S	Btu/lb.
100	0	67	0.7	16.7	1.3	81.3	1.18	—	1.6	98.4	1.43	—
95	5	68	1.1	15.8	1.8	81.3	1.17	—	2.2	97.8	1.41	—
90	10	72	1.6	14.8	2.0	81.6	1.04	—	2.4	97.6	1.24	—
85	15	72	1.4	14.9	1.8	81.9	1.07	—	2.2	97.8	1.28	—
80	20	73	2.1	14.4	2.0	81.5	1.11	11999	2.4	97.6	1.33	14370
75	25	74	2.4	13.5	2.2	81.9	1.06	—	2.6	97.4	1.26	—

*Average of three test samples, not adjusted for moisture.

ally beneficial to the macro-strength properties of coke produced from retort charges blended with fusain, despite the further increased proportion of fines. This effect may be a response to better distribution of the macerals in the charge, contributing to improved bonding, more uniform structure, and better accommodation of the volume changes developed during coking.

The effects of high proportions of fusain in the charge upon the chemical properties of the resultant cokes are shown in table 32 and figure 82. Increased proportions of fusain in the charge were accompanied by decrease in fixed carbon and ash in the coke. On the contrary, both moisture and volatile content of the cokes increased with the proportion of fusain; the sulfur trend was not emphatic but declined generally with increased fusain. Unfortunately before these results were obtained, all available fusain had been used to complete the blend series so that analyses of the original material could not be made.

EFFECTS OF "RESIN" OR "ASPHALTENE" ADDITIVE

In the course of studies upon certain of the coal seams of New South Wales undertaken in the Coal Research Laboratories of the Department of Geology and Geophysics, University of Sydney, Draycott (1954) demonstrated that a benzene-soluble, petroleum-ether-insoluble fraction of soft pitch, when added in small proportions to the coal charge, produced substantial improvement in the coking characteristics of the majority of the coals examined. The results of more comprehensive studies are being prepared for publication. The precise nature of the soft pitch extract is apparently unsettled, the terms "resin" and "asphaltene" having been used by various authorities.

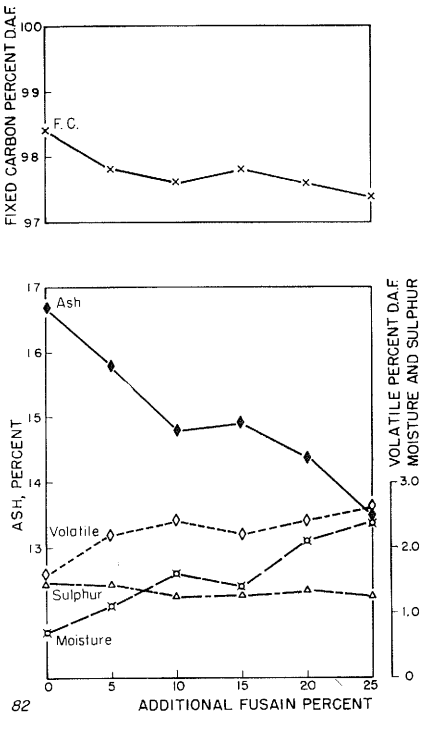
To investigate the possibility that such additives may improve the quality of coke produced from coal beds of Illinois, a series of studies was completed with the additive applied in various proportions to standard samples of both the No. 5 and No. 6 coals, as well as to fusain-coal blends of the latter seam.

PREPARATION OF THE "RESIN" ADDITIVE

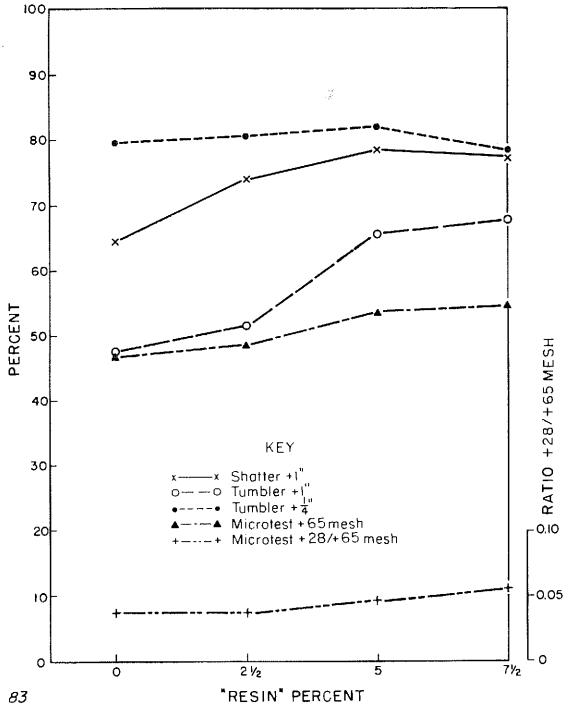
The bulk sample of soft pitch was supplied by the Inland Steel Company of East Chicago, Indiana. The procedure of extraction was substantially that described by Draycott (1954). One hundred grams of the pitch, broken into fragments no larger than 0.5 inch, were placed in a liter flask, to which was added 400 ml. of benzene; the mixture was refluxed on a steam cone for one to two hours. While hot, the digest was filtered into 4 liters of well stirred petroleum ether (40° to 60° range) and the insoluble residue in the flask was washed with 50 ml. of benzene which was also filtered into the petroleum ether. The precipitate which formed was collected by suction filtration, washed three or four times with petroleum ether, and dried at room temperature.

Twenty such runs gave a total yield of 586.5 grams representing 29.3 percent by weight of the pitch used. The "resin" or "asphaltene" extract was brown, finely granular or "fluffy," and readily developed a high electrostatic charge; this latter characteristic appeared to assist considerably in securing a uniform distribution when mixed with the coal charge.

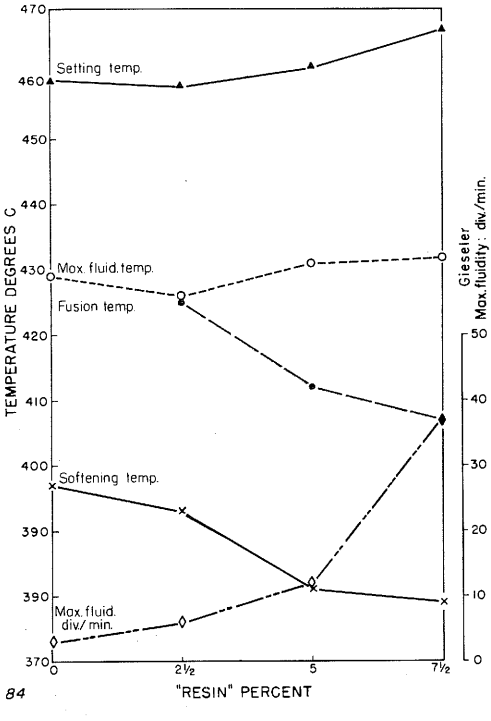
In the course of determining the melting point of the "resin" or "asphaltene" it gave evidence of decomposition, becoming shrivelled and blackened


FIXED CARBON PERCENT D.A.F.
F.C.
ASH, PERCENT
VOLATILE PERCENT D.A.F.
MOISTURE AND SULPHUR
Ash
Volatile
Sulphur
Moisture
ADDITIONAL FUSAIN PERCENT

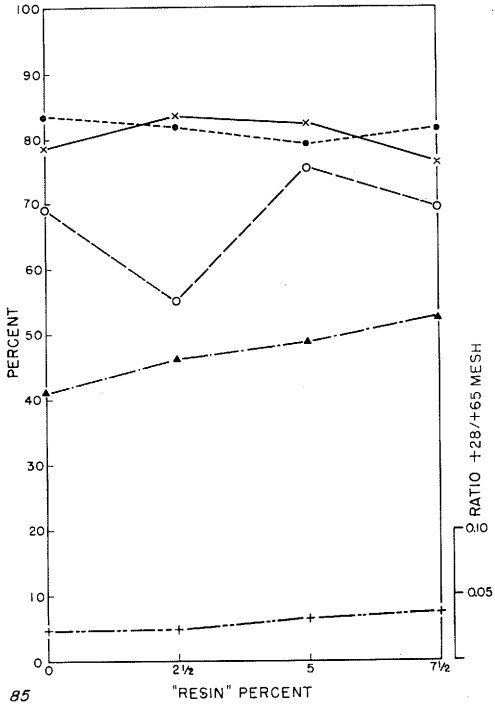
82


PERCENT
RATIO +28/+65 MESH
KEY
Shatter +1"
Tumbler +1"
Tumbler +¼"
Microtest +65 mesh
Microtest +28/+65 mesh
"RESIN" PERCENT

83


TEMPERATURE DEGREES C.
Setting temp.
Max. fluid. temp.
Fusion temp.
Softening temp.
Max. fluid. div./min.
Gieseler Max. fluidity: div./min.
"RESIN" PERCENT

84


PERCENT
RATIO +28/+65 MESH
"RESIN" PERCENT

85

at 120.5°C; it melted at 148.9°C. Chemical analysis returned:

Loss of weight at 107°C – 1.10%	
Composition	
Hydrogen	4.59
Carbon	92.35
Nitrogen	1.91
Oxygen (by difference)	0.75
Sulfur	0.40
Ash	0.00
Total	100.00

As the material could not be completely redissolved in benzene, the ebulloscopic method of molecular weight determination using benzene as a solvent could not be used.

Effects of the "Resin" Additive Upon No. 5 Coal

Samples of the reference pillar of the No. 5 coal, of standard size consist, were blended uniformly with "resin" in the proportions of 2½, 5, and 7½ percent and coked under standard conditions (table 33, fig. 83). All macro- and micro-strength indices of the resultant cokes were improved progressively and quite substantially when assisted by the additive in proportions up to and including 5 percent. The higher proportion of additive (7½ percent) in the charge was accompanied by a decline in tumbler hardness and shatter index, but the tumbler stability and micro-strength indices revealed further improvement. The luster of the cokes was enhanced although they appeared to be darker in color.

Gieseler values and free swelling indices were determined for each of the coal-additive blends (table 34, fig. 84). With increasing proportions of "resin" the softening and fusion temperatures appeared to fall considerably, but it is not clear to what extent this effect may be due to melting of the additive and consequent "lubrication" of the coal particles. The relatively low temperatures of decomposition and melting of the "resin" (120.5°C and 148–9°C respectively) could indicate a low coking point for the material and thus probably exclude the suggested effect of "lubrication."

The Gieseler maximum fluidity (dial divisions per minute) increased with additive proportions as did also both temperatures of maximum fluidity and setting with the exception of those of the mix containing 2½ percent "resin." Within the range of the conditions of experiment, the over-all effect of the additive appears to be an appreciable and proportionate extension of the plastic range of the coal charge and a relatively small increase in maximum fluidity. These effects, together with possible "lubrication," would assist in better "packing" of the charge, and consequent improved opportunity for "bonding" through the larger surfaces of contact between particles over an extended range of plasticity.

Fig. 82.—No. 5 coal: Influence of fusain on fixed carbon, volatile matter, moisture, sulphur, and ash content of cokes. Reference pillar, standard coking conditions (table 32).

Fig. 83.—No. 5 coal: Influence of "resin" additive on coke. Reference pillar, standard size consist, standard coking conditions (table 33).

Fig. 84.—No. 5 coal: Gieseler characteristics of reference pillar with "resin" additive for samples producing coke of Fig. 83 (table 34).

Fig. 85.—No. 6 coal: Influence of "resin" additive on coke. Special representative sample, standard size consist, standard coking conditions (table 35).

TABLE 33.—No. 5 COAL: INFLUENCE OF "RESIN" ADDITIVE ON COKE
(Reference pillar, standard size consist, standard coking conditions)

Resin added to standard reference pillar (percent)	Macro-test data of coke			Micro-test data of coke	
	Shatter: % + 1″	Tumbler		Mechanical strength test	
		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
0	64.6	47.5	79.4	46.9	0.037
2½	74.0	51.3	80.5	48.3	0.036
5	78.5	65.7	82.0	53.6	0.046
7½	77.2	67.8	78.2	54.5	0.055

TABLE 34.—No. 5 COAL: GIESELER PLASTICITY AND FREE SWELLING INDEX VALUES OF REFERENCE PILLAR BLENDED WITH "RESIN"

Sample, fraction and/or condition	Softening temp. °C	Fusion temp. °C	Maximum fluidity temp. °C	Setting temp. °C	Maximum fluidity div/min.	Free swelling index
Reference pillar	397	—	429	459	3	5
RP + 2½% "resin"	393	425	426	458	6	6
RP + 5% "resin"	381	412	431	461	12	6
RP + 7½% "resin"	379	407	432	467	37	6

TABLE 35.—No. 6 COAL: INFLUENCE ON COKE OF "RESIN" ADDITIVE, MINUS 150-MESH FUSAIN, AND REPRESENTATIVE SAMPLE BLENDS
(Standard size consist and coking conditions)

Special representative sample of standard size consist blended with		Macro-test data of coke			Micro-test data of coke	
		Shatter: % + 1″	Tumbler		Mechanical strength test	
Fusain −150 m.	Resin added		Stability: % + 1″	Hardness: % + ¼″	% + 65m	Ratio 28/65m
0%	0%	78.8	69.2	83.3	41.2	0.023
0	2½	83.4	55.0	81.9	46.1	0.024
0	5	82.2	75.6	79.1	48.9	0.032
0	7½	71.4	69.5	81.4	52.3	0.037
10	2½	87.7	81.7	85.0	48.0	0.038
20	2½	90.9	77.2	79.0	41.4	0.036
10	5	92.9	82.2	84.1	50.5	0.039
20	5	92.8	93.6	93.6	48.5	0.037

Effects of the "Resin" Additive Upon No. 6 Coal

As the supply of samples of the standard reference pillar of the No. 6 coal had been exhausted by the study series previously completed, a further large bulk sample (approximately 2,000 lbs.) of the coal was collected from the main haulage belt in the mine and used in the preparation of laboratory samples according to the standard procedure adopted for this project.

The "resin" or "asphaltene" additive, used in proportions of up to 7½ percent of the coal charge, induced progressive substantial increases in the micro-strength indices of the resultant cokes, a reflection of the development of a more resistant structure in the actual coke substance (table 35, fig. 85; pl. 9, A to D). The results of the macro-strength tests proved erratic. The shatter index of the coke produced was increased with 2½ percent additive, but there was no further increase when 5 percent was used, and it decreased with 7½ percent. Tumbler stability showed no clearly defined trend as related to the proportion of the additive introduced into the charge; tumbler hardness at first declined with increased "resin" and recovered only partially in the coke formed from the charge with 7½ percent additive.

Within the range of the present study upon the No. 6 coal the effects of the additive may be summarized as distinctly beneficial in relation to the micro-strength properties of the coke substance, but of doubtful or minor value in the improvement of macro-strength properties.

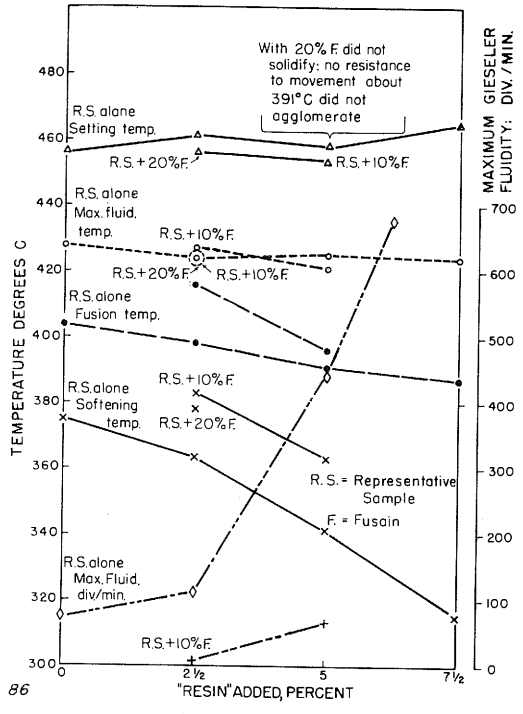
Gieseler determinations (table 36, fig. 86) revealed that, as in the case of the No. 5 coal, the effects of the additive had been to depress the temperatures of softening and fusion, to raise the setting temperature slightly, thus extending the plastic range; the maximum fluidity (dial divisions per minute) in this instance was increased quite substantially.

Effects of "Resin" Additive on Blends of No. 6 Coal and Minus 150-Mesh Fusain

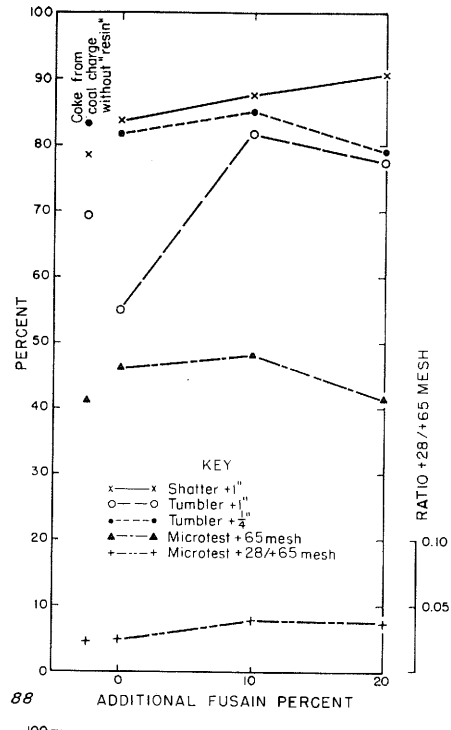
Although the effects of the "resin" or "asphaltene" additive had been beneficial in all respects when used in charges of the No. 5 coal, and of positive assistance in improving micro-strength and shatter characteristics of the cokes produced from No. 6 coal, there was evidence that greater benefits might result from its use in charges containing higher proportions of the fine size fractions or of material of otherwise inferior coking characteristics.

To investigate this possibility, different proportions of the "resin" were added to various blends of standard representative samples and fine fusain and the properties of the cokes produced from these "3-component" charges were compared with those of the normal coal alone, with the same "resin" content only, and as blended with fusain only (tables 35, 36, figs. 88, 89, 90; pl. 9, E to H).

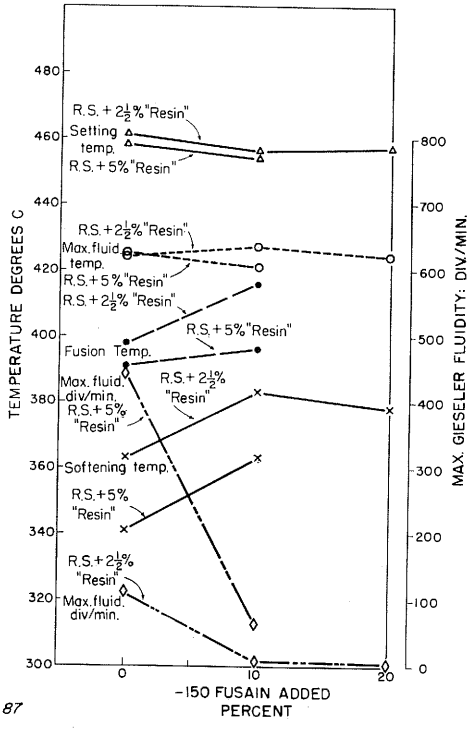
When the normal coal was blended with 10 percent additional fusain (minus 150-mesh) and 2½ percent "resin," all strength indices of the resultant cokes were increased in quite substantial amounts (fig. 88). Increase of fusain content of the charge to 20 percent with no more than 2½ percent "resin," reversed the trend in all but the shatter index. The great increase of fine, inert material had apparently been too great for the bonding powers of the low "resin" content.


With 20% F. did not solidify: no resistance to movement about 391°C did not agglomerate
R.S. alone Setting temp.
R.S. + 20% F.
R.S. + 10% F.
R.S. alone Max. fluid. temp.
R.S. alone Fusion temp.
R.S. alone Softening temp.
R.S. alone Max. Fluid. div/min.
R.S. = Representative Sample
F. = Fusain
TEMPERATURE DEGREES C
MAXIMUM GIESELER FLUIDITY: DIV./MIN.
"RESIN" ADDED, PERCENT

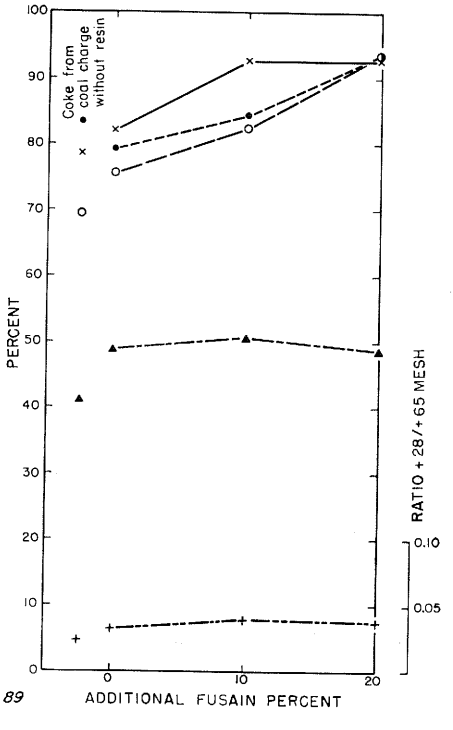
86


Coke from coal charge without "resin"
KEY
Shatter +1"
Tumbler +1"
Tumbler +¼"
Microtest +65 mesh
Microtest +28/+65 mesh
PERCENT
RATIO +28/+65 MESH
ADDITIONAL FUSAIN PERCENT

88


R.S. + 2½% "Resin" Setting temp.
R.S. + 5% "Resin"
R.S. + 2½% "Resin" Max. fluid. temp.
R.S. + 5% "Resin"
R.S. + 2½% "Resin"
R.S. + 5% "Resin" Fusion Temp.
Max. fluid. div/min.
R.S. + 2½% "Resin"
R.S. + 5% "Resin"
Softening temp.
R.S. + 5% "Resin"
R.S. + 2½% "Resin"
Max. fluid. div/min.
TEMPERATURE DEGREES C
MAX. GIESELER FLUIDITY: DIV/MIN.
-150 FUSAIN ADDED PERCENT

87


Coke from coal charge without resin
PERCENT
RATIO + 28/+ 65 MESH
ADDITIONAL FUSAIN PERCENT

89

TABLE 36.—No. 6 COAL: GIESELER PLASTICITY AND FREE SWELLING INDEX VALUES OF BLENDS OF "RESIN," MINUS 150-MESH FUSAIN, AND REPRESENTATIVE SAMPLE
(Standard size consist)

Sample, fraction and/or condition	Softening temp. °C	Fusion temp. °C	Maximum fluidity temp. °C	Setting temp. °C	Maximum fluidity div/min.	Free swelling index
Rep. sample	375	404	428	456	72	4
Rep. sample + 2½% "resin"	363	398	424	461	112	4
Rep. sample + 5% "resin"	341	391	425	458	444	6
Rep. sample + 7½% "resin"	315	387	424	465	676	5½
Rep. Sample 90% −150 Fusain 10% with addition of "resin" 2½%	383	416	427	456	7	5½
Rep. sample 90% −150 fusain 10% with addition of "resin" 5%	363	396	421	454	66	5
Rep. sample 80% −150 fusain 20% with addition of "resin" 2½%	378	—	424	457	3	3½
Rep. sample 80% −150 fusain 20% with addition of "resin" 5%	Did not solidify; no resistance to movement above 391°C. Did not agglomerate.					4

When the additive proportion was raised to 5 percent, blends of the standard coal and 10 percent fusain produced cokes which were improved to the same general degree in respect to tumbler stability, hardness, and micro-strength 28/65 ratio as when 2½ percent of "resin" had been used in the same blend; but shatter index and micro-strength 65 had been further and very appreciably improved (fig. 89). Increase of fusain content of the charge to 20 percent ("resin" remaining at 5 percent) was accompanied by considerable improvement of the cokes in both tumbler stability and hardness, these indices being the highest recorded in any test series of the entire project. The shatter index was maintained at the previous high level, but both micro-strength indices declined slightly. It would appear that the additional 2½ percent of "resin" permits the better "cementation" or "bonding" of the fine

FIG. 86.—No. 6 coal: Influence of "resin" additive on Gieseler characteristics. Representative sample and blends of representative sample with minus 150-mesh fusain (table 36).

FIG. 87.—No. 6 coal: Gieseler characteristics of representative samples blended with minus 150-mesh fusain and with "resin" additive (table 36).

FIG. 88.—No. 6 coal: Influence of "resin" additive on coke produced from blends of coal and fusain. Special representative sample (standard size consist) and minus 150-mesh fusain plus 2½ percent "resin" additive, standard coking conditions (table 35).

FIG. 89.—No. 6 coal: Influence of "resin" additive on coke produced from blends of coal and fusain. Special representative seam sample (standard size consist) and minus 150-mesh fusain plus 5 percent "resin" additive, standard coking conditions (table 35).

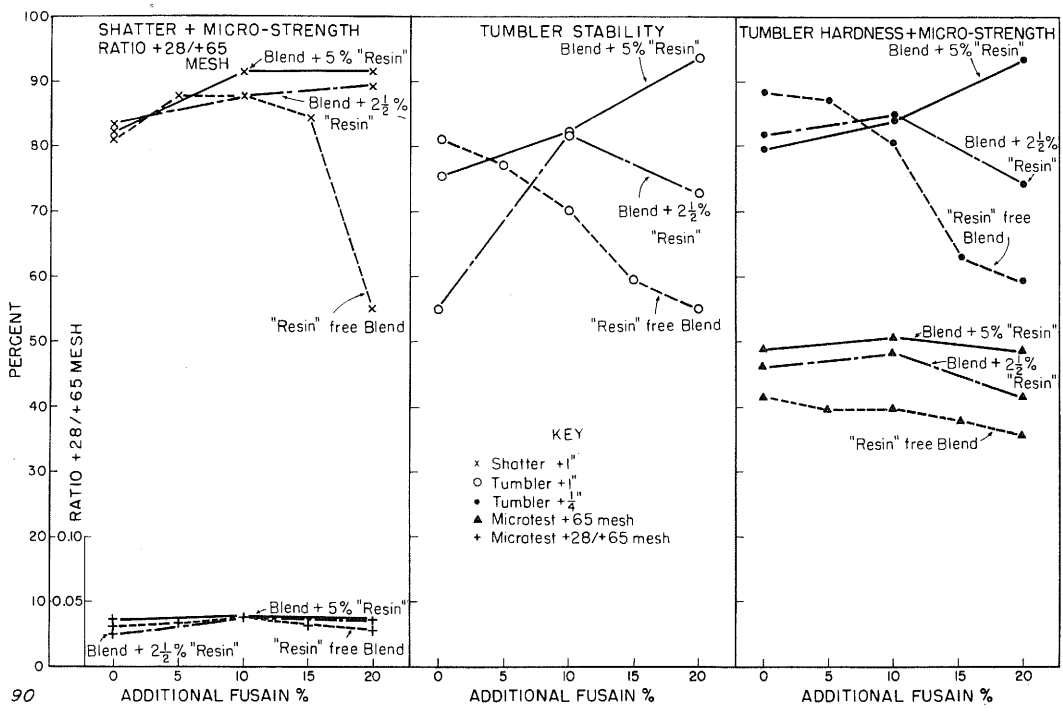


FIG. 90.—No. 6 coal: Influence of "resin" additive on cokes. Comparison of effects of varying proportions of "resin" upon blends of representative seam sample (standard size consist) and minus 150-mesh fusain, standard coking conditions (table 35).

fusain with the normal coal consist, thus more effectively accommodating the volume changes induced during coking to produce a coke of exceptional macro-strength. The slight decline in strength of the coke substance (as shown in the micro-strength indices) with the further increase of 2½ percent of additive may be due to a slight excess of "resin" in the "interstitial" coke produced by the cementation of the fine size fractions.

The effects of the "resin" additive may perhaps be best appraised by direct comparison (fig. 90) of the mechanical properties of cokes produced from normal coal blends with fine fusain, without and with the addition of the additive in different proportions. The progressive relative variation and contrast in the properties is particularly striking.

The effects of the additive and of the fine fusain in modifying the Gieseler characteristics of the coal charge may be gauged by reference to figures 86 and 87. Increased proportions of fusain raise the temperature of softening and fusion, but lower the setting temperature and maximum fluidity and reduce substantially the plastic range of the charge. With the addition and increase in the proportions of "resin" additive, even in the fusain blends, the maximum fluidity is greatly increased, the softening and fusion temperatures are considerably reduced, and the setting point is maintained more or less constant; the plastic range is significantly extended.

SUMMARY AND CONCLUSIONS

The petrographic constitution of a coal importantly influences its coking characteristics. The evidence is particularly significant in view of the limited petrographic variability of each of the coal seams studied and the preliminary nature of the investigation.

Compromise techniques of coal sample preparation, of coking, and of coke testing were adopted which, although not directly comparable with pilot plant results, nevertheless gave results and established trends which may be indicative of those of pilot plant or larger scale.

Detailed petrographic analysis of the maceral proportions and dimensions in the selected macro-type and seam sections, both as regards over-all characteristics and those of the various size fractions, demonstrated a general and systematic variation in petrographic composition which is indicative of preferential breakage of the coarser coal-type bands and constituents. Systematic variability was discovered by broken coal analyses of coal macro-types, the seam sections and the size fractions of each of the standard samples. Limited correlation was possible between petrographic, chemical, fluidity and swelling characteristics of both samples and fractions.

Exploration of the influence of the "thermal environment" during coking was limited to those factors which could be readily assessed and reasonably controlled with the resources available. The maximum uniform rate of temperature increase of which the oven was capable happened to be about the same as that which appears to prevail in the greater part of the charge in pilot scale and industrial ovens, and therefore was accepted as standard (3.6°C per minute).

By charging standard samples at different temperatures and completing the coking process at a standard final temperature (1010°C) for an identical period (two hours), the broad effects and importance of initial temperature of charging in relation to the plastic range of the charge were established (450°C for No. 6 coal; 540°C for No. 5 coal).

Coals charged at temperatures below or just within the plastic range yielded cokes of greatest macro-strength as determined by shatter and tumbler tests; coals charged at temperatures significantly higher than the upper limit of the normal plastic range produced cokes in which the macro-strength was greatly decreased but the micro-strength considerably improved. The latter property was also generally found to improve with higher temperatures and longer periods of final coking; both these factors tended to affect adversely the macro-strength characteristics of the cokes.

From the results of these introductory coking experiments, it was possible to define standard conditions of coking for each coal. The results also demonstrated the desirability of a much more exhaustive investigation of the thermal environment in relation to the plastic properties of the charge with particular reference to rates of heating and temperature increase below, within, and above the plastic range.

The effects of coal type (with reference to maceral proportions, dimensions, and distribution) upon the character of the coke were considerable despite the limited nature of the type variation. They could be explored effectively for only the No. 6 coal. In general, the proportions and distribution of vitrinite in the charge appeared to be

important, the optimum concentration in the No. 6 coal under the laboratory conditions of coking being approximately 87 percent with a median particle size of 15 microns. There emerged also a slight but definite trend towards increase of coke strength with greater content of inertinite, which showed no indication of changing at concentrations of the order of 9 percent.

In the test series on individual size fractions the coking characteristics could not be effectively related to inherent petrographic or chemical variation. The substantial variation which these cokes demonstrated could be most reasonably explained as a function of the size characteristics of the original size fraction; the 35 x 65 mesh size range appeared to be critical in relation to both macro- and micro-strength. Despite their individually poor coking qualities, the importance of the finer fractions in the development of coke of optimum quality from a charge of "balanced" size consist was clearly demonstrated.

In view of the high proportion of fines which is either lost during commercial preparation or deliberately discarded, the results of this study are provocative. More especially is this true when it is considered that the fines are generally relatively enriched in fusain, which the results of these study series indicated as being of potentially considerable importance in the control of coke quality and generally not present in fully adequate proportions in bright coal.

The influence of over-all coal size consist upon coke quality was found to be substantial. In general the minimum of disintegration or shattering during preparation, obtained by controlled coal breakage, screening of undersize and re-circulation of oversize within a closed system, gave the best size consist for the production of cokes of optimum mechanical properties. The size distribution was simple, progressive, and "balanced" and constituted the standard size make-up.

The basis of size consist designation for graphic analysis of the results of this study series is not considered wholly satisfactory but it was possible to establish a relatively simple relation between size consist in the charge and character of the coke produced. Although difficult to assess, it is considered that of the comparatively few anomalous cokes which appeared in the course of these studies, the majority could be attributed directly or indirectly to the effects of bad size distribution within the individual coal charges. Care was taken to avoid this, but efforts were not always successful.

The addition of inertinite or finely divided fusain (minus 150-mesh) in appropriate proportions to the coal charge of standard size consist always improved some of the strength indices. Other values remained constant, declined progressively, or improved after an initial decrease. In general, the improvement was concerned principally with the macro-strength characteristics and could be further emphasized by proper adjustment of the size consist of the coal charge. When recrushed at 1/16 inch the standard samples produced very little increase in fines but a substantially greater proportion of the medium sizes; blended with fusain the macro-strength increased substantially but the micro-strength decreased more rapidly. Almost comparable improvement in shatter index and tumbler stability, but an accelerated decline in the micro-strength indices, accompanied

further crushing of the charge at 1/32 inch.

Standard type or seam samples containing high proportions of vitrain provided excellent cokes when blended with fine fusain in proportions of up to 15 percent. The effects were enhanced when the size consist of the oven charges was reduced by recrushing at 1/16 inch. The upper seam section of the No. 5 coal (that which is normally mined) produced particularly good cokes when blended with as much as 15 percent additional fine fusain; both macro- and micro-strength indices showed substantial improvements over the normal coke.

The results of the studies of coal-fusain blends in which high-vitrain content and increased proportions of the medium size fractions emerged as particularly significant factors, demonstrate the importance of both petrographic composition and thorough mixing in the coal charge. They are also in complete accord with the trends established on the basis of coal-type and maceral variation in relation to coking properties.

Of more than casual interest is the behavior of the mineral matter (dominantly fine sedimentary material) of the lower seam section of the No. 5 coal which appears to behave as an "inorganic inertinite" with a correspondingly marked improvement in the mechanical properties of the coke. The unexpectedly high quality of the metallurgical cokes produced from some of the relatively high-ash coals of Australia and India may be at least partly attributable to the same factors.

The brief investigation of the influence of "resin" or "asphaltene" additives upon the character of the laboratory cokes, demonstrated clearly that they were of limited value when used in normal oven charges of coals which already possess quite well defined coking properties. Upon charges of the coal-fusain blend series the beneficial effects of the additive were quite remarkable and offer possibilities of important application in coking coals with an undue proportion of either fines or "inerts."

In conclusion, it must be emphasized that although the results of these studies may be more widely applicable to coals of characters comparable with those of the No. 5 and No. 6 beds of Illinois, individual investigation probably would be essential to effective utilization.

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