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The electron microscope is being used in the examination of soil clays.

IN THIS ISSUE

Electron Microscopy of Soil Clays and Related Materials

BY THE PHYSICAL RESEARCH BRANCH,
BUREAU OF PUBLIC ROADS, AND
THE NATIONAL BUREAU OF STANDARDS

Reported by EARL B. KINTER,[1]
Highway Physical Research Engineer,
ADOLPH M. WINTERMYER,[1]
Highway Physical Research Engineer,
and MAX SWERDLOW,[2]
Physicist

The importance of soil properties in the design, construction, and maintenance of highways has made their measurement and evaluation of primary concern to the engineer. Wide recognition that much of the physical behavior of soils stems from their clay constituents has long indicated the need for a better fundamental knowledge of these complex materials. Basic research on the soil clay-mineral content offers the most logical approach to this goal. Electron microscopy provides a visual means for investigating the size, shape, and structure of the minute particles present in soil clays and is an aid in identifying the constituents. This article reports an exploratory examination made with an electron microscope of selected clay minerals, soil clays, and related materials. Objectives of the study were to make a general examination of a broad variety of soil clays to provide basic data for subsequent correlation with information obtained from other analytical methods and performance tests, and to estimate the potential usefulness of this research tool in soils engineering work.

IN RECENT YEARS, investigators of the properties of soil materials and their application to highway work have become increasingly concerned with the constituents of soil clays. It is definitely known that clay substances, even when present in relatively small quantities, exert a strong influence on soil properties. An estimate simply of the amounts of clay contained in soils is of limited application, however, since two soils having the same approximate clay content frequently possess wide variations in properties. It has been evident that the clay fractions themselves are inherently different, and that a better understanding of soils and their properties and uses should result from an increased knowledge of the nature and properties of the constituent substances of soil clays.

Although the study of clays has been going on for many years, efforts have been seriously handicapped for want of direct and adequate scientific tools. Within the past two decades, however, considerable advances have been made through the application of modern techniques. For instance, it has been established that earthen clay materials, including the clay portions of soils, consist chiefly, although not entirely, of minute crystalline particles of hydrous aluminum silicates (13)[3]. These have been called the clay minerals.

A number of individual members of three principal clay mineral groups or families have been identified, and many details of crystal structure and its relation to their properties are well agreed upon. In general, however, this field of study remains in a rapidly growing stage, from both the fundamental and the practical aspects.

Of the methods applied to the problems of identifying and characterizing the constituents of clays, those of X-ray diffraction, differential thermal analysis, specific surface area measurements, and chemical analysis (including base-exchange studies) have been especially important. In all these methods the investigator is faced with many difficulties arising from the heterogeneous and impure nature and the small particle size of the material under study. Each method has supplemented the others and not one of them has afforded complete characterization. A further and quite different means of attack has been made available by the advent of the electron microscope, which, through its high resolving power, has made possible the advantages of visual examination of individual soil clay particles.

The first application of electron microscopy to clay minerals was made in Germany (10) and, after 1940, was extended in the United States (9, 15, 18 19). Examinations of certain soils with the electron microscope have been reported (6, 16, 17, 22, 24), but an intensive application to a large number of soils has apparently not heretofore been made. This report describes a preliminary study in which a number of soil clays, clay minerals, and related materials were examined with an electron microscope.

The study was of an exploratory nature, with two main objectives: (1) a general examination of a broad variety of soil clays with respect to their particle size, shape, structure, and identification; and (2) an estimate of the potential usefulness of the electron microscope in soils engineering work.

Materials Examined

A selection of 95 soil clays, clay minerals, and associated materials was examined in the microscope and micrographs were obtained. The soils, most of which are referred to by accepted soil names, cover a wide range of conditions of soil formation and parent materials. In most cases, they were subsoils containing relatively little organic matter. Many of them were from stock samples of the Bureau of Public Roads, and a few were from private collections of the authors.

The minerals examined represent almost all of the individual members of the clay-mineral groups having generally accepted mineralogical names, and a small number of non-clay minerals likely to occur in soil clays. Where available, two or more examples of the individual clay minerals were included. These were obtained from Dr. Ralph E. Grim of the University of Illinois, the American Petroleum Institute, several mineral supply houses, the Harbison-Walker Refractories Company and other commercial suppliers of clays and ceramic materials, the National Museum, and the collections of the authors. In view of the difficulties involved in obtaining authentic

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[3] Italic numbers in parentheses refer to references listed on page 100.

specimens, especially of the clay minerals, the authors wish to express their sincere thanks to all those who made samples available.

The particle size limits of the clay fraction of soils are not unanimously agreed upon. In the Bureau of Public Roads classification, 5 microns (0.005mm.) are taken as the maximum diameter of clay, and the fraction below 1 micron (0.001 mm.) is referred to as colloid material. The U. S. Department of Agriculture and the International Society of Soil Science designate clay as that fraction below 2 microns. In any case, for visual considerations, clay particles are well below the range of the unaided human eye, which cannot distinguish between resolved objects separated by a distance smaller than 200 microns (0.2 mm.). Under optimum conditions, the absolute limit of resolution obtained with the light microscope is 0.2 micron (0.0002 mm.). For practical purposes, however, its effective limit is much above this.

The Electron Microscope

One of the fundamental factors that limit the resolving power of any microscope is the wavelength of the radiant energy illuminating the object. Theory and experiment demonstrate that about one-half of the wavelength of the illuminating source sets an absolute and unalterable lower limit to the inherent resolution obtainable. In contrast to the relatively long wavelengths of visible light, the electron microscope utilizes the extremely short wavelengths associated with a beam of electrons. Although the theoretical limit of resolution has not been achieved, the electron microscope, since its inception in 1932, has been developed to the practical limit of resolving power—about 20 Angstroms ($1A = 10^{-4}$ micron = 10^{-7} mm.). This represents a hundred-fold advantage in resolving power over that of the light microscope.

Figure 1 illustrates the functional components of the light microscope and the magnetic-type electron microscope. In the light microscope, lens L_1 serves to focus the illumination from its source (a lamp, in this case) on the subject S. The objective lens L_2 forms a real, inverted, enlarged image I_1. From this first image the eyepiece lens L_3 forms a virtual, inverted, greatly magnified image I_2.

In the electron microscope, as seen in figure 1, a hot cathode emits a cloud of electrons, a portion of which is accelerated through an electrostatic field and through the anode aperture to form a divergent beam of electrons. The first magnetic coil L_1 serves as a condensing lens to collimate the beam which is directed on the subject S. Rays penetrating the subject are focused by a second magnetic coil L_2 to form an enlarged real image I_1, a section of which is further enlarged by the third magnetic coil L_3 to form a final real image I_2 on a fluorescent screen or a micrograph on a photographic plate.

Although the components of the two instruments fulfill similar functions, such phenomena as polarization or color are not possible with the electron microscope. Transmitted electrons form real images that can be viewed on a luminescent screen or photographically recorded in black and white. In electron microscopy, entirely new methods for preparing specimens and different concepts for interpreting the observed electron-optical images are required. Because of the poor penetrating power of electron beams, only small and very thin objects can be examined and, further, the examination must be made with the object under high vacuum.

Preparation of Samples

Samples for this study were prepared as dilute suspensions by dispersing a small quantity of untreated material in distilled water in a Waring Blendor, after a preliminary soaking period of an hour or more. In some cases, a drop or two of dilute ammonia water was added before stirring. The suspension was separated into the desired particle-size ranges by the customary centrifugal method. Although a thorough study of particle-size distribution and frequency was not undertaken at this stage of the work, certain separations of the soil-clay fractions were made, including the following size ranges: 10 to 1 microns; less than 2 microns; less than 1 micron;

Light Microscope   Electron Microscope

Figure 1.—Functional components of the transmission light microscope and the magnetic electron microscope.

and less than 0.2 micron. Separation of the 10-to-1-micron fraction was discontinued after a few preparations because most of the particles were too massive and opaque (nontransparent to the electron beam) for adequate examination (fig. 7D) and obviously bore little relation to the clay fraction.

The 1-micron separation was also rejected because its upper limit frequently proved to be too low to include all the clay-mineral content. The separations containing particles smaller than 0.2 micron were examined in some soils but usually appeared as a finer counterpart of the coarser clay. In many cases, material which was obviously not one of the hydrous clay minerals was noted to occur down through the range of this fraction. Finally, separation at 2 microns was found to be most satisfactory for the general examination desired in this work, since it included virtually all of the clay-mineral content and, near its upper limit, a representation of particles of the more rock-like minerals common to the nonclay fractions. For a more detailed investigation of the finer clay particles, separations other than those employed here would be both desirable and necessary.

After dispersion and centrifugation, the samples were further diluted with distilled water to make a faintly cloudy suspension which was stored in a small, capped vial until used for preparation of the microscope specimens. Further dilutions were made later in the vials as found to be necessary. Because of the large dilutions required, the heterogeneous nature of the materials studied, and the high magnifications obtained, very careful controls were maintained throughout all the procedures in order to prevent contamination. To prepare specimens for the electron microscope, a very thin film of Formvar (polyvinyl formal) was cast on a clean water surface and transferred to a one-eighth-inch disk of 200-mesh, stainless-steel screen. A drop of the sample suspension was placed on the plastic film and allowed to settle and dry in an air-conditioned room (72° F., 60 percent relative humidity). At this stage, the specimen was ready for examination.

Shadowcasting Technique

The RCA type EMU (50 kv) electron microscope was used. The direct magnification of this instrument ranges from about 1,000 to 25,000 diameters, with a possible useful magnification of about 250,000 diameters obtainable through optical enlargement of the micrographs. In this study it was found most suitable to use about 6,000 diameters as the original electronic magnification, with subsequent optical enlargement to give a total linear magnification of about 21,000. The 1μ line on the photographs illustrating this report represents 1 micron. In most cases, at least five 2-inch-square micrographs were made of characteristic fields of each material examined. In all, about 1,250 electron micrographs were taken.
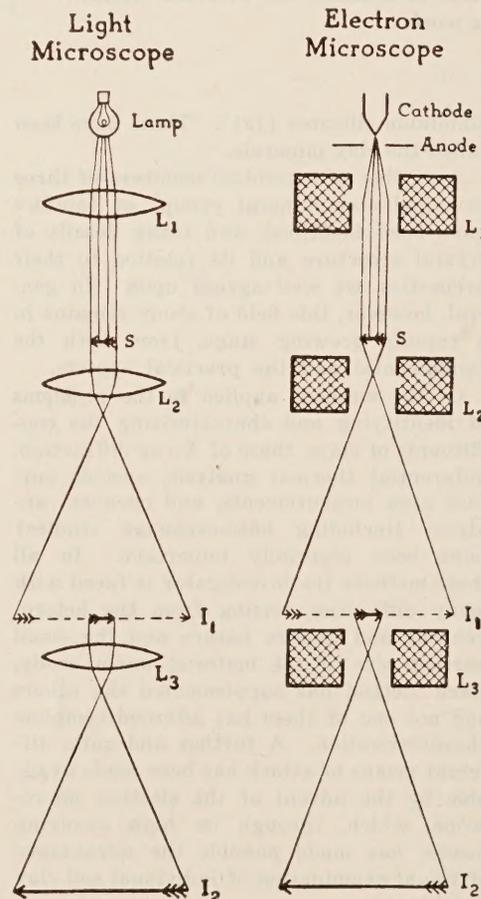
Since metallic shadowcasting (*23, 24*) adds contrast as well as a three-dimensional aspect to surface details, a very thin coating of evaporated chromium metal was deposited obliquely upon the dispersed and mounted specimens. This was accomplished by placing the dried specimens in a vacuum chamber at a fixed angle and distance from a tungsten-wire basket containing about 75 mg. of pure chromium. When the pressure in the vacuum chamber reached 10^{-5} mm. of mercury, the tungsten crucible was electrically heated, causing chromium vapor to radiate in all directions. As the atoms condensed, a very thin film of metal was deposited on the specimens. The sample particles are elevations behind which are shielded areas (shadows) receiving no metallic coating. In the microscope, such areas readily transmit the incident beam of electrons, whereas the thicker and chromium-coated areas scatter the electron beam and less of the incident energy is transmitted.

Thus, from the length of the "bright" shadows, as observed in the electron microscope, relative heights of the particles and details of surface structure can be deduced. Positive photographic prints are shown in all of the illustrations in order to permit direct comparison between shadowed and unshadowed specimens. These represent the images as seen in the microscope. Black indicates material opaque to the electron beam; white indicates transparent material. Shadow length is approximately four times the altitude of the particles.

Results and Discussion

Electron micrographs of several soils and clay minerals are presented in figures 2–7. These show only a few of the many samples examined, and were selected to illustrate some of the more evident points of information available through the use of the electron microscope. In a random scanning of a specimen, marked differences are encountered, as illustrated in the two micrographs of a sample of Manor clay (figs. 7A and 7B) and those of Illinoian glacial drift (figs. 7C and 7D). Thus, a single micrograph or view, as used in this report, is not necessarily representative of the whole material under discussion but is, however, of value in pointing out certain features of the sample. On the other hand, in the actual examination a rapid survey of the whole specimen, or usually of several specimens, permits a quick comparison of the general morphology of the material and a rough estimate of the relative proportions of different constituents, even though they have not been fully identified. Where possible, the most representative field was chosen for the micrograph.

Kaolins

In soils work, when clays are mentioned, the kaolins first come to mind. In fact, clay formation has long been referred to as "kaolinization." This is the result of the early location and use of kaolin deposits and the belief that kaolin was the chief constituent of soil clays. Although kaolin may be the predominant clay mineral present in some soils, it is now understood that it may be only a minor component or entirely absent in other soils. Originally assumed to be a single mineral, it is now recognized as a family or group of clay minerals, of which kaolinite is the leading member.

Crystals of a well-formed kaolinite are shown in figures 2A and 2B as thin, six-sided plates. The perfection of crystal outline varied considerably among the several kaolinites examined—in some cases, only an occasional straight side or 120-degree angle could be found, although the thin, plate-like characteristic was common to all specimens. It may be noted that some of the crystals are elongated, whereas the faces of others are more nearly equidimensional. Stacking and thinness of the plates are evident, both from their transparency and from the length of shadows. Most of the plates lie in the plane of the plastic substrate but the "book" of kaolinite crystals, in the upper part of figure 2A, illustrates an aggregate with the plates nearly at right angles to the film. The relatively great depth of field of the electron microscope is demonstrated in areas of figure 2A, where settling of kaolinite particles upon each other during the drying of the specimen has superimposed several plates of various sizes and thicknesses.

This feature is of exceptional value in making visible the details of a wide range of particle sizes and thicknesses at a single focusing adjustment. These relatively pure kaolinites, English china clay, and Georgia kaolin, were formed as residual deposits from granite, apparently under conditions favorable to good crystalization. On the other hand, most of the commercial kaolin clays examined were much less perfectly formed and relatively less pure.

Halloysite, seen in figures 2C and 2D, is characterized by tubular-shaped crystals, as indicated by the greater transparency of their hollow centers. Split tubes and concentric layering may also be observed. In earlier examinations of halloysite, the particles were thought to be lathlike (*2*), but later studies confirmed the hollow, tubular nature (*3, 5, 6, 9*). This crystal form is in sharp contrast to the six-sided plates of kaolinite, despite the fact that both minerals have the same chemical composition, approaching $Al_2O_3 . 2SiO_2 . 2H_2O$. Although the existence of cylindrical characteristics has been revealed, the exact mechanism of the growth of tubular crystals has not been explained. Nevertheless, the marked differences in the morphologies of these two kaolins must have a profound influence on the physical properties of soils containing these minerals.

With regard to the other members of the kaolin family, dickite showed a platy habit very similar to that of kaolinite, and allophane consisted of irregular, angular masses with a glassy appearance. Nacrite was not examined in this study. It has been reported to resemble kaolinite and dickite in form, but with particle sizes as large as 5 mm. (*3, 9*).

Although micrographs of allophane have been published (*9*) in which the particles were shown as spherical masses, our observations were not in agreement with this. We noted that the particles were angular at the start but, after a short exposure to the electron beam, were sufficiently melted to produce the globular shapes. Compared to halloysite and kaolinite, the minerals dickite, nacrite, and allophane are relatively rare and are not thought to occur widely in soils.

Montmorillonites

Although the identity of montmorillonite as a mineral was established many years ago, it was not at first regarded as being a part of soil clays. More recently, however, it has been shown to be widespread in soils. The term montmorillonite is now used to designate the clay-mineral family as well as its chief member. Among the other members are nontronite, beidellite, and hectorite. The familiar trade term bentonite has been applied to adsorptive clays and drilling muds but properly refers to a clay rock consisting essentially of the sodium-rich montmorillonite or, less often, beidellite clay mineral. The origin of bentonite is considered to stem from the devitrification and chemical alteration of glassy igneous material. In soils, however, the montmorillonites may be either water laid or formed residually from a variety of parent minerals.

As shown in figures 2E, 2F, and 3A–D, the appearance of the members of the montmorillonite family is in decided contrast to that of the kaolins. The montmorillonite member (fig. 2E) is characterized by loose, fluffy floccules or aggregates of indefinite shape and size. The nebulous outlines and the variable translucency indicate aggregates formed from much smaller and thinner units. These features seem to be correlated with the magnitude of surface area required for the high base-exchange capacity, large volume changes, and the strong bonding and plastic properties manifested by the montmorillonites. Beidellite (fig. 3B) is similar in appearance to the montmorillonite member, as are the bentonites (figs. 2F and 3A). Nontronite, the iron-rich member, is seen in figure 3C as long, thin fibers which have a tendency to remain in bundle-like aggregates, or sometimes as long, flat, ribbon shapes. Hectorite, (fig. 3D) is in the form of very thin needles or fibers, shorter than those of nontronite, with a pronounced tendency towards a more random arrangement. When not completely dispersed, hectorite crystals are arrayed in thin, lath-like aggregates.

The crystal lattice structure of the montmorillonites would indicate the occurrence of platy particles. Although montmorillonite, beidellite, and the bentonites are platy,
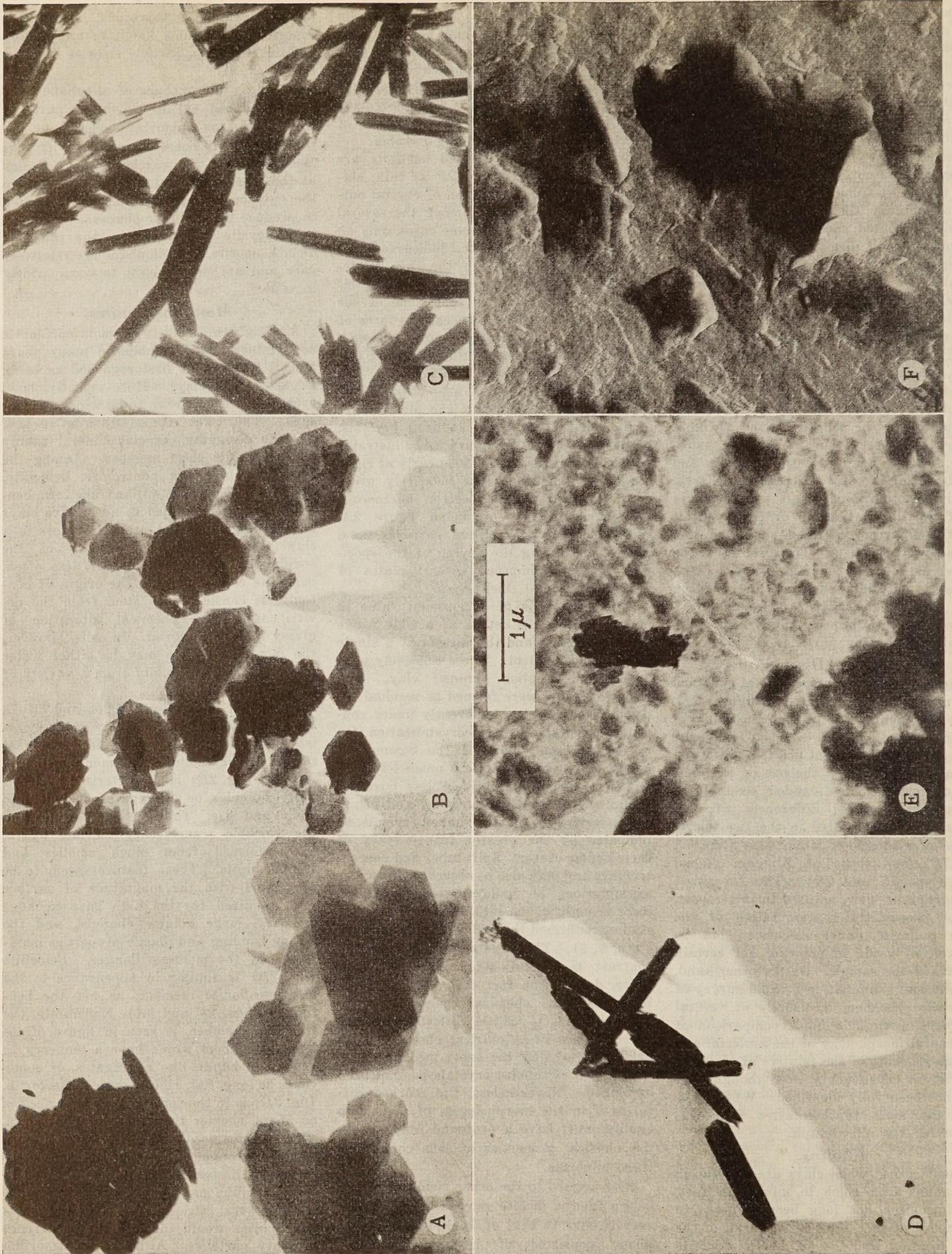
Figure 2.—*A, English china clay (kaolinite); B, Georgia kaolin; C, D, halloysite, Utah Lake, Utah; E, montmorillonite, Otay Mesa, Calif.; F, sodium bentonite, Wyoming (electron micrographs; total electronic and optical magnification = 21,000 diameters).*

1 μ
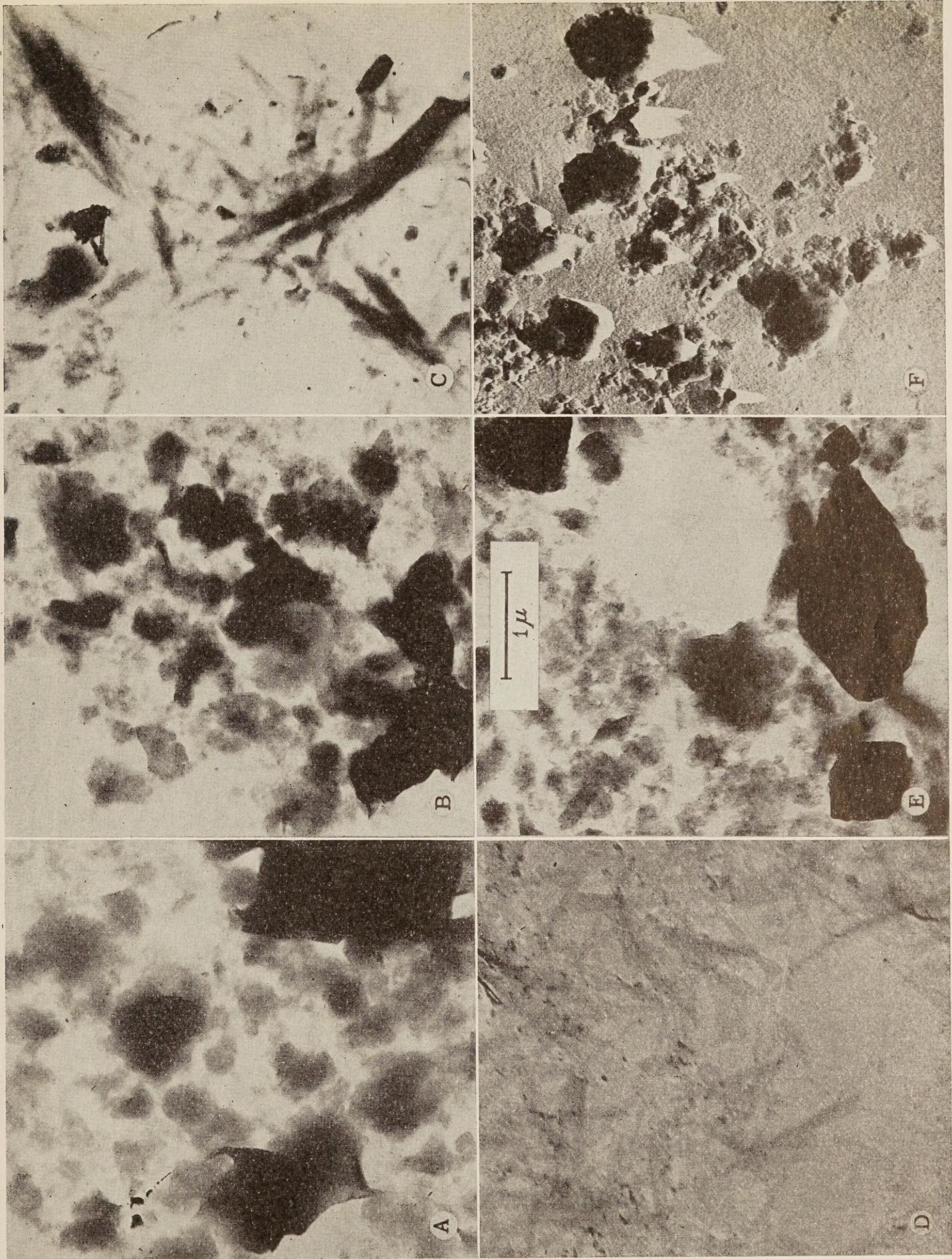
Figure 3.—*A, calcium bentonite, Mississippi; B, beidellite, San Diego County, Calif.; C, nontronite, Manito, Wash.; D, hectorite, Hector, Calif.; E, illite, Morris, Ill.; F, illite, Fithian, Ill. (electron micrographs; total electronic and optical magnification = 21,000 diameters).*
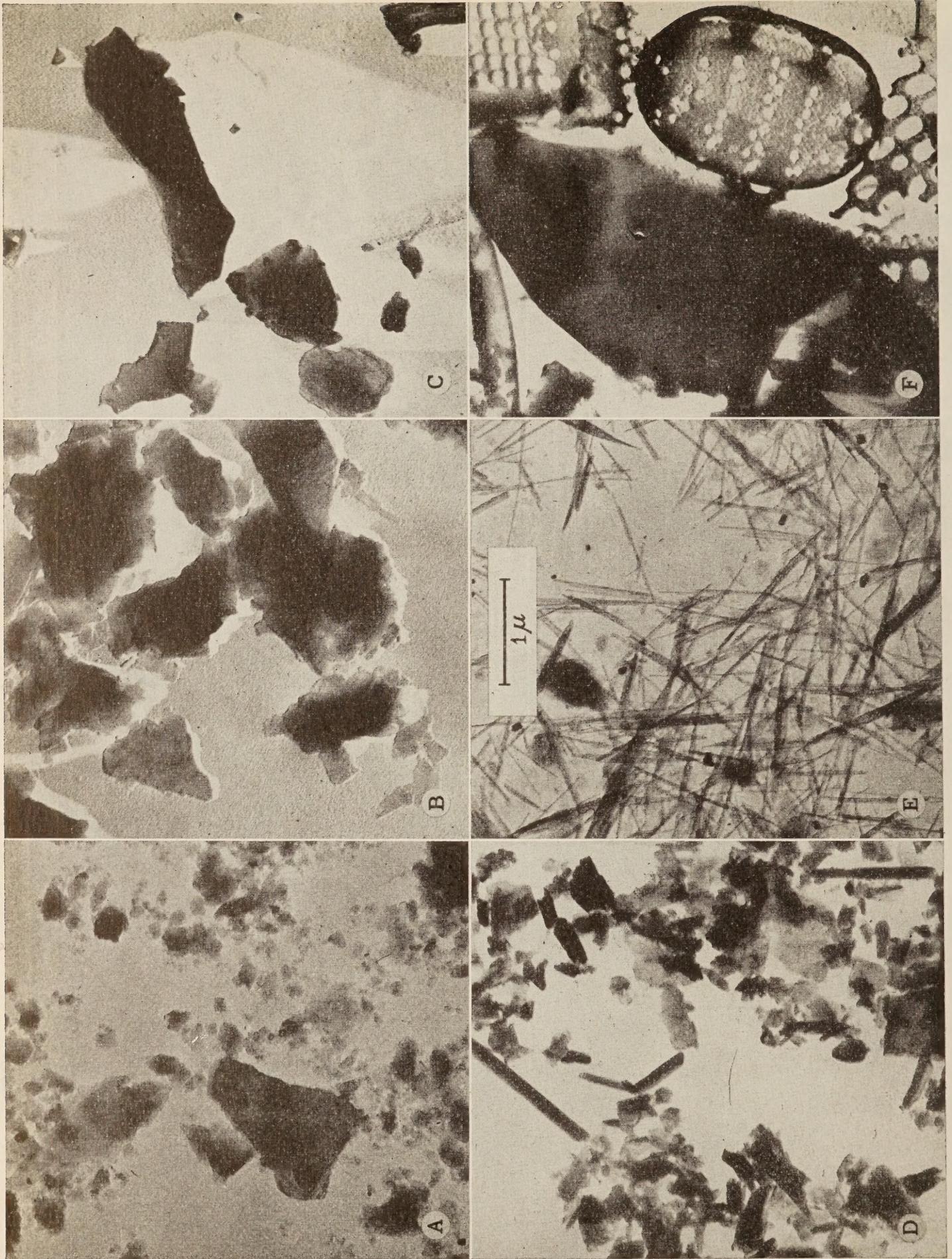
Figure 4.—A, illite, Fithian, Ill.; B, metabentonite, Tazewell, Va.; C, silica (quartz), Ottawa, Ill.; D, mica, Spruce Pine, N. C.; E, attapulgite, Attapulgus, Ga.; F, diatoms, Santa Barbara County, Calif. (electron micrographs: total electronic and optical magnification = 21,000 diameters).
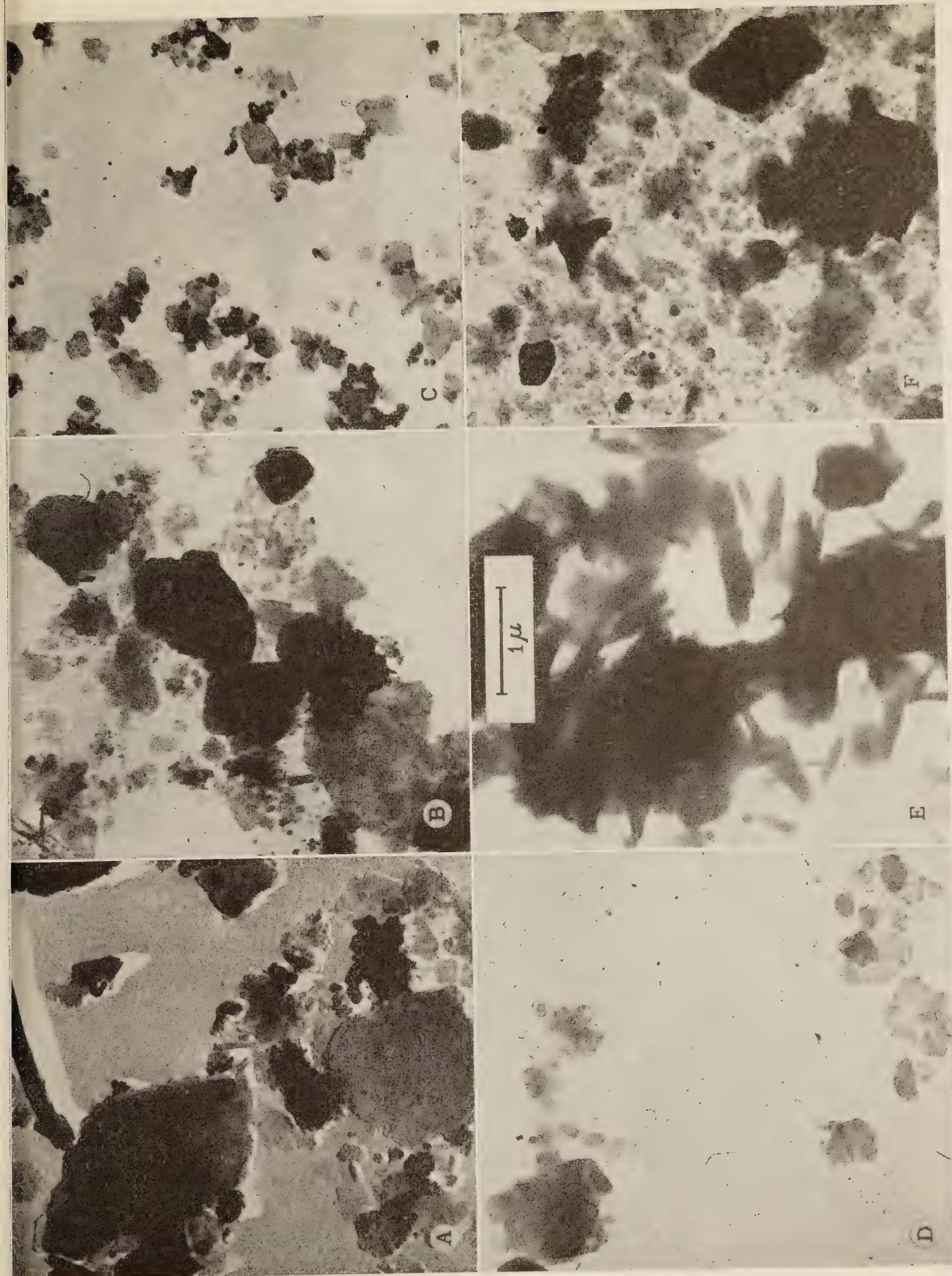
Figure 5.—A, Cecil clay; B, Tuxedo clay; C, Philadelphia red clay; D, Philadelphia white clay; E, Houston clay; F, Putnam clay (electron micrographs: total electronic and optical magnification = 21,000 diameters).
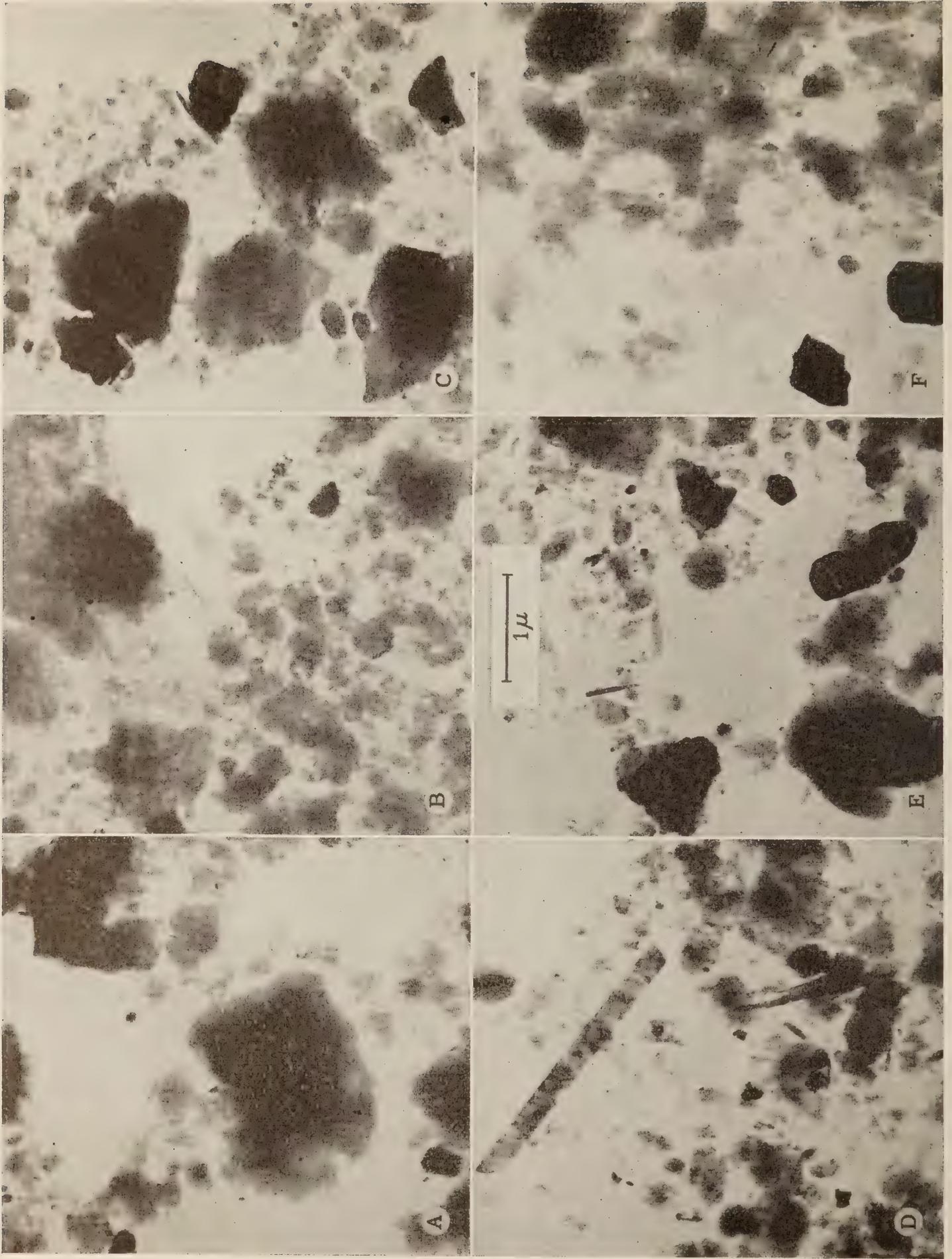
Figure 6.—A, Iredell clay; B, Miami clay; C, Crosby clay; D, Gila clay; E, Hagerstown clay; F, Wabash clay (electron m...
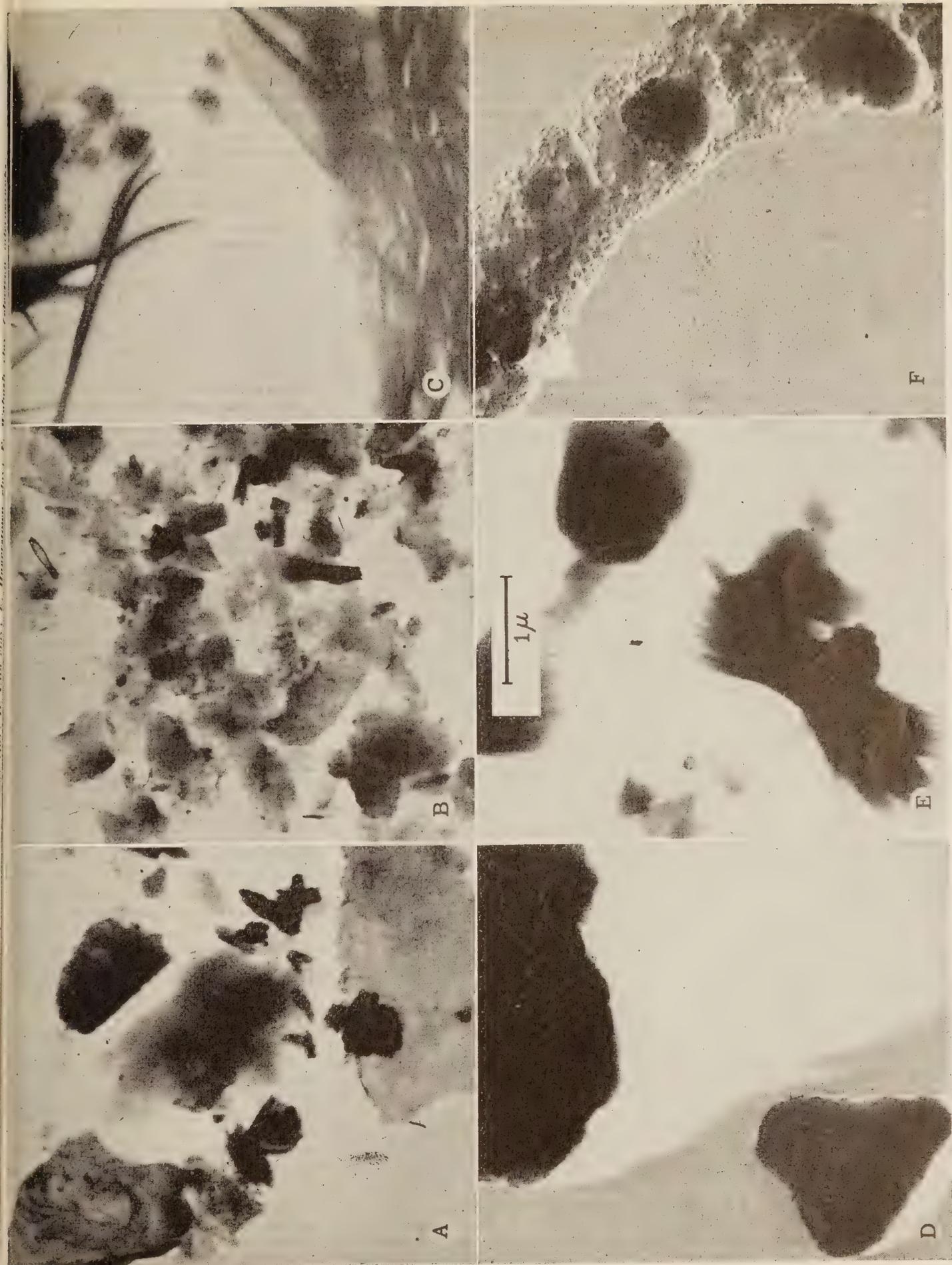
Figure 7.—A, B, Manor clay; C, D, clay from Illinoian glacial drift; E, Marshall clay; F, Pullman clay (electron micrographs: total electronic and optical magnification = 21,000 diameters).

this is not the case with nontronite and hectorite which exhibit a crystal habit resembling needles or fibers. The occurrence of nontronite in the ribbon-like form may be attributed to the greater atomic radius of the ferric ion in the crystal lattice, resulting in a strain restricting the width but permitting growth along the length to produce fibrous crystals (21). The long narrow crystals of hectorite are so thin and transparent as to be almost indistinguishable individually.

Illites

The illite or hydrous mica group of clay minerals is not as well known and understood in some respects as are the two groups previously discussed. The name illite is used as "a general term for the clay-mineral constituents of argillaceous sediment belonging to the mica group" (11); specific characterization into separate species within the group is still to be accomplished. Illite is reported as a common constituent of slates, shales, and soil clays (4, 11, 12).

As seen in figures 3E, 3F, and 4A, illite particles are vague and indefinite in shape, with little resemblance to crystalline material. However, plates or flakes with rough but relatively sharp boundaries may be observed. It may be noted that some of the illite material closely resembles the fluffy habit shown by montmorillonite. This feature makes difficult, if not impossible, a positive distinction between these two materials. In general, however, illite presents a denser and more definitive appearance than montmorillonite. Flakes with relatively sharper outlines and frequently of fairly large diameter are also more common to illite. These are most likely remnants or relics of partly altered mica. The two euhedral masses seen at the bottom of figure 3E are thought to be iron pyrite; the large irregular mass between them is probably quartz.

Related or Allied Minerals

Many of the non-clay minerals likely to be associated with soil clays were observed to bear little resemblance to any of the clay minerals. In general, they were dense masses with no distinct morphologies. However, some of them, such as mica, attapulgite, and diatomaceous earth, have distinctive features and are easily recognized. Particles of ground silica (fig. 4C) were, as expected, quite fragmental, with sharp points and knife-like edges. The platy habit of finely divided mica is seen in figure 4D. Although this sample was represented as a very pure mica, it contained a considerable amount of a rod-shaped impurity in addition to the mica sheets. These were different from the narrow strips which mica yields as a result of secondary cleavage perpendicular to the plane of the sheets, as shown at the top of figure 6D. It may be noted that some of the thin, nearly transparent mica sheets in figure 4D show uneven bands resulting from interference or

diffraction phenomena between the layers. During the manipulation of the microscope controls to bring various fields of the specimen into focus, these bands frequently were seen to move across the mica sheets. This movement was thought to be the result of heating and electrical charging, with the effect of driving off water from between the sheets or buckling of the sheets themselves. This latter effect, although useful in the examination, could not be photographed.

The acicular structure of attapulgite, a fuller's earth from Attapulgus, Ga., is seen in figure 4E. Although, in general, this needle-like form appears similar to that of sepiolite and serpentine, the crystals of attapulgite are characteristically shorter and straighter than those of other fibrous minerals.

Part of a sample of a diatomaceous earth is shown in figure 4F. This material consists chiefly of the siliceous remains of diatoms, a large class of microscopic algae which contribute to deposits derived from both fresh and sea water.

Examination of Soil Materials

The clay minerals are for the most part the chief constituents of soil clays. It has been clearly recognized that the kind and amount of clay in a soil strongly affect its suitability as an engineering material and thus have a direct bearing on the design, construction, and maintenance of highways and other structures. The foregoing discussion and the interpretations and concepts derived from the electron micrographs of the pure clay minerals cannot, however, be expected to delineate fully the nature of soil clays. The genesis of soils is a dynamic process, characterized by a colloidal system involving a large number of free ions and the hydrous oxides of silicon, aluminum, and iron. Varied parent material, weathering conditions, and dynamic organic processes, along with the vigorous operations of dislodgement, transportation, and deposition, combine to produce a soil environment that, in general, makes the soil clay a heterogeneous body with chemical and physical properties quite at variance with those of any single, pure, well-formed clay mineral. A study of the mineral content of soil clays, however, in conjunction with studies of the clay minerals themselves, provides a logical approach to the complex mechanisms relating such factors as origin, chemical alteration, crystalline structure, and variable constitution of these principal constituents whose physical and chemical behavior are so strongly reflected in the properties of the soil body.

A selection of electron micrographs of several of the soils examined is presented in figures 5-7. As previously noted, these do not comprise a comprehensive coverage of all possible soil conditions, but are presented as a random view of what may be seen in the examination of a selection of soils from many different areas and back-

grounds of soil formation. The mixed [na]ture of soil clays is a point immediat[ely] brought out by most of the micrograp[hs] in direct contrast to the relatively grea[ter] purity of the mineralogical specimens. F[or] the most part, the tubes of halloysite rema[in] relatively unchanged in form, as do a[ll] other rod or needle-like materials, but we[ll] formed crystals of kaolinite as six-si[ded] plates are almost entirely absent.

Soils Containing Kaolinite

Examples of soils in which kaolinite [is] the predominant clay mineral are given [in] the micrographs of Cecil (fig. 5A) a[nd] Tuxedo (fig. 5B) subsoils and of Philad[el]phia red clay (fig. 5C). Cecil is a residu[al] soil formed from granitic rocks in t[he] southern part of the Piedmont soil provin[ce] just west of the Atlantic Coastal Pla[in.] Tuxedo and Philadelphia red clay are wate[r] laid, sedimentary materials common to t[he] Coastal Plain itself. The latter, taken fro[m] a deep boring in Philadelphia, was we[ll] mottled with white areas, a common effe[ct] among poorly drained subsoils. Parts [of] the white areas were used to prepare t[he] micrograph, in figure 5D, of Philadelph[ia] white clay. In these several figures, kao[li]nite appears as the major component, wi[th] an admixture of opague (dark) round[ed] material.

In the natural state, each of these soi[ls] is strongly colored by red oxides of iro[n,] probably hematite, which may be the opaq[ue] material seen in the micrographs. Th[e] mottling apparently is the result of abru[pt] changes in the iron-oxide content as th[e] white clay contains very little of this in[g]redient and is mostly pure kaolinite.

Most of the particles seen in these sample[s] are quite distinct in outline, with shar[p,] although irregular, edges. In the case [of] Tuxedo clay (fig. 5B) there is, howeve[r,] a considerable quantity of small-graine[d,] poorly defined material which may provi[de] a clue to the reasons for the known highl[y] plastic nature of this clay, since kaolini[te] alone is usually found to be only weakl[y] plastic. Although electron microscopy doe[s] not provide an absolute identification of thi[s] material, the value of such an examinatio[n] is clearly demonstrated in the indication [of] its presence so that the aid of other method[s] may be applied to its further study an[d] possible identification.

Other Soil-Clay Constituents

Montmorillonites have been well-establ[ished] lished (1, 13, 21) as common constituents o[f] soil clays in varying proportions to othe[r] clay minerals. Of the several soils examine[d] in this study, examples in which montmoril[lo]lonites are predominant are shown in figure[s] 5E, 5F, and 6A. These include Houston cla[y] (8, 20), from the Gulf Coastal Plain o[f] Texas; Putnam (22) from a loessial area o[f] Missouri; and Iredell (21), a residual soi[l] derived from diabasic rocks in the souther[n] Piedmont. Iredell (fig. 6A) appears to con[tain]

sist largely of a single, relatively pure substance closely resembling the nebulous floccules of the mineral specimens of montmorillonite.

Putnam, shown in figure 5F, contains beidellite (21, 22)—very similar in appearance to the montmorillonite member. In addition, there is a non-clay constituent ranging in size down to a small fraction of a micron. It may be noted here that both magnesium-saturated and natural Putnam soils were used in this work, as were also examples of other soils having sodium and calcium as the adsorbed cations, but that little difference in morphology, except perhaps in the mode of flocculation, could be ascribed to the difference in cations. In the case of the sodium and calcium bentonites shown in figures 2F and 3A, respectively, the floccules exhibit warping and resemble a cornflake shape.

Examples of Miami, Crosby, Gila, and Hagerstown soils, reported (1, 7, 8) to be high in illite content, are shown in figures 6B, 6C, 6D, and 6E. Miami (fig. 6B) is widespread in the North Central States and is derived from calcareous glacial till. The transparent, smaller particles are believed to be illite in association with darker fragments of non-clay minerals. Some of the larger particles may also be illite or aggregates of illite and montmorillonite, which is also present (7) in Miami soils. The presence of rod and needle-like crystals and many small dark fragments, in figure 6B, illustrate the mixed composition not unexpected in a material derived from glacial action.

A comparison of figure 6B, of Miami clay, and figure 6C, of Crosby clay, shows the close similarity of the clays of these two soils, which are both members of the Miami soil catena and are derived from the same parent calcareous glacial till under different conditions of slope and drainage.

This is of interest in that electron-microscopial examination may assist in the correlation of soil types and in pointing out similarities or differences between two examples of the same soil obtained from different areas. For instance, the colloidal content of these two soils from Indiana has been reported (7) to consist chiefly of hydrous mica (illite) with a smaller content of montmorillonite. Although their mineralogical compositions were closely similar, they were different in thermal decomposition and base-exchange behavior. Other work on the clay of a Miami soil from Wisconsin (8) found montmorillonite predominant and illite as a minor constituent. In cases of this sort, a more detailed study of specific separations of the various constituents might prove helpful.

Gila clay, from a desert soil of Arizona, derived from granitic and gneissic rocks, is seen in figure 6D to contain an abundance of illite (8) as well as a rich admixture of non-clay minerals. Note the strip of mica in the upper-central portion of the micrograph. Hagerstown clay (fig. 6E) also appears to consist largely of illite. This soil is formed residually in limestone areas of the Eastern and Central States. Samples of Hagerstown clay from Maryland and Missouri have been reported (1) to contain about equal proportions of kaolinite and illite. The sample used here, obtained from Monroe County in Indiana, apparently contains both of these along with other minerals.

Supporting Information Needed

It is believed that montmorillonite and illite may be associated in mixed layer aggregates (9). The metabentonite from Tazewell, Va., shown in figure 4B may be a clay mineral of this sort. Such occurrences are even more probable in soil clays, as in Wabash clay (fig. 6F), an alluvial soil reported to contain both of these minerals. Such intimately mixed, layered aggregates of extremely fine-grained particles cannot be clearly differentiated and identified by electron microscopy alone.

The need for supporting information is especially evident where opaque masses largely lacking in distinctive features are present, as well as where some soil-clay constituents are unidentifiable yet have more specific morphologies. For example, the fibrous material in figure 7C, resembling sepiolite, was observed in a sample of Illinoian glacial drift but cannot be positively identified from the micrographs alone. Sheaves of very fine fibers, possibly nontronite, were found in the clay extracted from Marshall silt loam, a prairie soil from the loessial area of western Iowa (fig. 7E) as well as in Houston clay (fig. 5E). A gel-like material, possibly silica gel, is shown in figure 7F, covering and binding together the more discrete particles of Pullman clay, a soil occurring extensively in the Texas Panhandle. The effect of such gels on the properties of soils is probably of considerable importance but as yet has not been evaluated. Certainly, however, their presence is discernible only by a method permitting direct visual observation in the colloidal range. Soils derived from mica-bearing parent material may also contain micas in the clay fraction. Similarity of the platy habit of mica and kaolinite make their differentiation difficult when the crystals are poorly developed, small in size, and fragmented and aggregated, as in Hagerstown clay (fig. 6E).

Summary

In the study of materials so small in particle size, complex in nature and properties, and varied in composition as soil clays, a single method cannot possibly provide a complete understanding of the many factors involved. The use and value of X-ray diffraction, differential thermal analysis, and chemical-analytical approaches are well-established. Electron microscopy can be very helpful in identifying and characterizing some of the constituents of soil clays and in providing a rough estimate of their relative proportions. For constituents not possessing specific morphological features, it can provide complementary clues and a background for information obtained from other methods.

Particle size and the relative distribution of particle-size ranges are well-known to be important in considerations of soil properties. These are no less important in the case of the soil-clay fraction itself. In a correlation of the amount of clay or of its quality to the behavior of a soil, separation must be made at some arbitrary figure for the upper particle-size limit. In estimating the effect of different sized particles on the behavior of the soil, however, consideration must be given to the particle sizes found in the whole soil sample as well as in the clay fractions obtained therefrom. In the clay size range, such considerations are helped in a direct way by electron microscopy, in which particle size and shape can be observed and semiquantitative estimates of the relative amounts of size ranges may be made.

Having once examined a variety of soil clays with the electron microscope, one is convinced that any detailed investigation of similar materials by other methods alone would be severely handicapped for want of visual examination. Moreover, the application of surface-replica techniques and the further development of other methods of specimen preparation will likely be of value in studying the structure of both consolidated and natural soils and the effect of additives in the amelioration or control of soil properties. In its own right, the electron microscope provides valuable information about soil clays which can be obtained in no other way. Admittedly, this essential concept has been well recognized by those closely allied to this research tool. It was felt, however, that the methods of electron microscopy should be specifically directed to the problems associated with soils as engineering materials. And it is from this point of view that this preliminary report has been made.

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A Cooperative Study of
Fillers in Asphaltic Concrete

BY THE PHYSICAL RESEARCH BRANCH
BUREAU OF PUBLIC ROADS

Reported by CARL A. CARPENTER
Senior Highway Research Engineer

ONE of the important problems that requires the attention of highway, materials, and research engineers is that of evaluating the mineral aggregates that may be potentially available for use in highway base and surface construction. The judicious use of local aggregates and of various byproducts is an important means of reducing construction costs.

In Massachusetts, the quarrying and processing of large quantities of traprock for various purposes, including highway construction, has resulted in the accumulation of large waste piles of traprock fines which, so far, have not been marketable and therefore constitute an expensive and troublesome disposal problem.

In the hope that a use might be found for at least a part of this material and with a view to reducing bituminous surfacing costs to some extent, a cooperative research project [1] was initiated to determine, by laboratory tests, an indication of the possible value of traprock dust as a filler for asphaltic concrete. Investigation was also made of the value of fly-ash fillers, obtained as a byproduct from the combustion of powdered coal.

It was agreed that the evaluation of the proposed fillers should be made by using them in a standardized asphaltic concrete which would be prepared, molded into test specimens, cured, and tested under carefully controlled standard conditions and procedures.

Conclusions

The first two of the following conclusions relate to the resistance to water of mixtures containing the fillers under investigation. Attention is called to the fact that other possible effects of the fillers were not in-

The judicious use of local aggregates and of mineral byproducts in highway construction can effect considerable savings in costs, through lower purchase prices and lesser transportation charges. Large waste piles of traprock fines, accumulated at quarries, and fly ash, a residue from combustion of powdered coal, offer cheap sources of fines for use in bituminous surfacing if they are found to be satisfactory for the purpose.

The cooperative study reported here was initiated to determine, by laboratory test, an evaluation of such materials as fillers in asphaltic concrete. In this type of pavement, a satisfactory filler aids in resisting the softening action of water on the asphalt-aggregate mixture. It was found, in these tests that, for the materials tested, traprock dust would give satisfactory, and fly ash superior, resistance to water in bituminous concrete paving mixtures of the dense type. Other effects of these fillers were not investigated, and it is possible, of course, that they may be of some importance.

From this study it was found that the 4-day, 120° F. immersion of the standard immersion-compression test generally can be relied upon to differentiate between satisfactory and unsatisfactory fillers. For border-line cases, a 7- or 14-day immersion may be necessary to clear up any doubt.

Where determination of compressive strength or stability is important, the study showed that test specimens must be molded from freshly prepared mixtures, without any reheating or reprocessing. This restriction is apparently unnecessary when the only information desired is the effect of water on the compressed mixture.

vestigated and it is possible that, in some cases, these may be of importance.

The results of this laboratory study have been consistent in indicating that traprock dust of the type furnished by New Jersey and Massachusetts can be expected to give satisfactory resistance to water when used as a filler in bituminous concrete paving mixtures of the dense type.

The laboratory studies indicated consistently that the fly-ash fillers, represented by those from New Jersey and one from Illinois, can be expected to give superior resistance to water in bituminous concrete paving mixtures of the dense type.

The tentative standard immersion-compression test,[2] requiring immersion at 120° F. for 4 days, generally can be relied upon to differentiate between satisfactory and unsatisfactory fillers. For obtaining this differentiation, the criterion of 75-percent retention of the dry strength, which was suggested in connection with earlier studies,

appears to have been further substantiated by the data in this report since the fillers that failed to meet it were those that, on the basis of reported experience, would be expected to give poor results in the field. However, further correlation with field experience seems desirable to establish more firmly the lowest permissible percentage of retained strength.

In cases where fillers, although passing the 4-day immersion test, are suspect because they are border-line cases or because of high silica content, extension of the immersion period to 7 or 14 days may be resorted to in order to clear up any doubt.

Where measurement of the compressive strength or stability of the mixture is the main consideration, it is important that the test specimens be molded from freshly prepared mixtures without reheating or reprocessing of any kind, since it has been found that both reheating and reworking—as, for instance, breaking up pavement samples and remixing—cause substantial increases in the apparent compressive strength.

[1] The cooperators were the laboratories of the Massachusetts Department of Public Works, represented by A. V. Bratt, Chief of Laboratory, who originally proposed the study; the New Jersey State Highway Department, represented by Fred H. Baumann, Chief, Testing Division; the Ohio State Department of Highways, represented by R. R. Litehiser, Chief Engineer, Testing and Research Laboratory; the National Crushed Stone Association, represented by A. T. Goldbeck, Engineering Director; and the Bureau of Public Roads, represented by E. F. Kelley, Chief, Physical Research Branch. This report has been reviewed and approved by all the cooperators.

[2] Tentative method of test for effect of water on cohesion of compacted bituminous mixtures. A.S.T.M. designation D 1075–49T.

When the only information desired is the effect of water on the compressed mixture, it appears from past experience and from the data in this report that the caution against reheating or reworking need not be observed because the increase in strength caused by these factors is approximately proportional in the dry and wet tests and, therefore, the percentage of strength retained is not appreciably affected.

Basis of Study

The principal test characteristic upon which the ratings of the fillers were based was the resistance of the compacted asphaltic concrete mixture to loss of strength after immersion in water at 120° F. for 4 days. The procedure for making this test, commonly called the immersion-compression test, was that outlined in a paper before the Association of Asphalt Paving Technologists [3] and later adopted in two parts as tentative standards [4] by the American Society for Testing Materials. Briefly, the test for a given mixture consists of subjecting a set of three companion specimens of the compacted mixture to an unconfined compression test to determine the "dry" compressive strength. A duplicate set of three specimens, prepared at the same time as the first, is immersed in water for 4 days at 120° F. and this set is then tested for "wet" compressive strength. The percentage of the original or "dry" compressive strength that is retained in the "wet" test is reported as the retained strength. With only such exceptions as will be noted in connection with the test data, the specimens for both the dry-strength test and the immersion test, in these studies, were cured in air for 24 hours at 140° F. after molding as required in the standard procedure.

Crushed traprock for coarse aggregate, and glacial sand for fine aggregate, as well as two filler materials—traprock dust and commercial limestone dust—were furnished for the first series of tests by the Massachusetts laboratory.

At about the time the initial work was getting under way, the suggestion was made that a similar study of fly ash, a waste product of coal-fired, electric power plants, be made a part of the project [5]. The laboratories of Massachusetts, New Jersey, Ohio, and the Bureau of Public Roads agreed to take part in this second phase of the investigation.

Additional quantities of crushed traprock and glacial sand, as well as a sample of silica dust, were supplied by the Massachusetts laboratory and two samples of filler, one a silica dust and the other a fly ash, were supplied by the New Jersey laboratory. Silica dust was included as a check or control material because of its known, generally poor, behavior as a filler.

Upon completion of the second phase of the study, a third series of tests was proposed in which sufficient new samples of traprock dust and fly ash were to be tested to corroborate fully the indications obtained in the two previous series. An additional coarse aggregate, rhyolite from Massachusetts, was brought into the study and rhyolite dust made from this same material was also included, together with two traprock dusts and three additional fly-ash samples that were supplied by the New Jersey laboratory. Other coarse aggregates and fillers were included in the work of some of the individual laboratories as will be indicated by the data in the tables and discussions.

Master Grading Used

In the original plans, a master grading was worked out and was used throughout the three phases of the study. Because of variations in the apparent specific gravity of the different materials used, the only way that a constant particle-size distribution could be maintained was to reduce all of the proportions in the master grading to an absolute-volume basis. For this purpose, the absolute volume of 7 parts by weight of limestone dust per 100 parts of total aggregate was taken as the basis for all the filler contents throughout the three series of tests. Thus, the variations in the amount by weight of filler shown in the various mixtures that will be described reflect only the variations in the apparent specific gravity of these fillers. Similarly, when coarse aggregate other than the original crushed stone was used, the proportion by weight was adjusted to give the same absolute volume of coarse aggregate as before.

In the first series, the bitumen content was 6½ parts by weight to 100 parts by weight of basic aggregate. In the second series some of the mixtures contained 6½ parts and some 5½ parts of asphalt per 100 parts of aggregate and in the third series, 5½ parts of asphalt were used for all mixtures with two exceptions which will be discussed in connection with the tabulated data.

It was originally intended that the test mixtures should contain between 6 and 7 percent voids when compacted in order that the variable effect of water due to differences in the filler might be clearly defined. It was found, however, that the type of filler affected the density and, therefore, the void content, to a very considerable degree. No attempt was made to adjust out these differences because it was felt that they reflected some of the inherent differences in the fillers that should be brought out in the test results.

The basic grading that was used throughout the filler investigation is shown in table 1, which also shows the asphalt ratio that was used in the first series and part of the second.

The proportions by weight of the two test mixtures used in the first series of tests are shown in table 2. The mixture containing traprock dust shows the adjustment in the weight of the filler to obtain the correct absolute volume. The mixture containing limestone dust is, of course, the basic mixture shown in detail in table 1.

[3] *Application and present status of the immersion-compression test*, by J. T. Pauls and J. F. Goode, 1947 Proceedings of the Association of Asphalt Paving Technologists. See also, *Further developments and application of the immersion-compression test*, by the same authors, PUBLIC ROADS, vol. 25, No. 6, Dec. 1948.

[4] A.S.T.M. *Standards*, 1949, Part 3. Test methods D 1074–49T and D 1075–49T.

[5] This phase of the study was suggested by Mr. Fred H. Baumann, Chief, Testing Division, New Jersey State Highway Department.

Table 1.—Basic grading and mixture proportions for mixture containing limestone dust

	Parts by weight			
	Traprock	Sand	Limestone dust	Combined
Sieve sizes:				
½ inch to No. 4	38			38
No. 4 to No. 10	20	5		25
No. 10 to No. 40		19		19
No. 40 to No. 200		11		11
No. 200			7	7
Total aggregate	58	35	7	100
Asphalt, 85–100 penetration				6.5
Total, parts by weight				106.5

Table 2.—Proportions of the test mixtures in series 1

	Parts by weight of mixture with filler of—	
	Limestone dust	Traprock dust
Aggregate retained on No. 200 sieve	93	93
Filler	7	7.48
Total aggregate	100	100.48
Asphalt	6.5	6.50
Total	106.5	106.98

Table 3.—Proportions of the test mixtures in series 2

	Parts by weight of mixture with filler of—					
	Limestone dust [1]	Traprock dust [1]	Silica dust			Fly ash (N. J.)
			Md.	Mass.	N. J.	
Aggregate retained on No. 200 sieve........	93	93	93	93	93	93
Filler........	7	7.48	6.70	6.87	6.75	6.57
Total aggregate	100	100.48	99.70	99.87	99.75	99.57
Asphalt, series 2a......	6.5	6.5	6.5	6.5	6.5	6.5
Mixture totals, series 2a........	106.5	106.98	106.20	106.37	106.25	106.07
Asphalt, series 2b......	5.5	5.5	5.5	5.5	5.5	5.5
Mixture totals, series 2b........	105.5	105.98	105.20	105.37	105.25	105.07

[1] Same fillers as used in series 1.

The proportions by weight of the test mixtures of the second series are shown in table 3.

In planning the third series of tests, it was agreed that each cooperating laboratory would carry out only as many of the tests as could be done without interference with other work, and also that some of the laboratories might properly diverge from the group effort to conduct special studies on materials primarily of local interest. Thus, a great many material combinations were tested in the third series but relatively few of these were tested by more than one of the cooperators. A list of the material combinations of series 3 that were investigated by one or more of the cooperating laboratories is given in table 4. One of the cooperating laboratories made several additional mixtures which were used in exploring the effect of other methods of curing than those agreed upon and used by the group as a whole. The data from these extra tests are not reported here because there is no basis upon which they can be compared with the data obtained by the other cooperating laboratories.

Traprock Dust a Good Filler

The results of the tests on the two materials of series 1 are shown in table 5. Here the comparison is only between one sample of limestone dust and one sample of Massachusetts traprock dust. The four laboratories that participated in this first series showed good agreement on the percentage of original strength retained by the test mixtures after immersion. All of the results agreed in indicating that, as determined by this test, the traprock dust was equal to limestone dust.

The results of the cooperative tests on the mixtures of series 2 are shown in table 6. The New Jersey silica dust, a material already existing in the deposit as a natural powder and produced from the natural deposit primarily for the glass industry, was reported to have given good results as a filler in bituminous surfacing. This would seem to make it an unusual example of silica dust which, as a rule, is not favored

as a filler on the basis of its service record. This material showed good performance in the laboratory tests of series 2a, with high bitumen content. In series 2b, with low bitumen content, one sample gave good results and one gave poor results.

The Maryland silica dust, while showing good performance in the high-bitumen mixtures (6½ parts), was definitely inferior in those containing 5½ parts of bitumen per 100 parts of aggregate.

At the conclusion of the series 1 tests, it had been suggested that the bitumen content of 6½ parts might be high enough to mask the effect of poor quality in certain fillers and it had been agreed that two pro-

Table 4.—Material combinations tested in series 3 [1]

Mixture No.	Coarse aggregate		Fine aggregate		Filler (dust) [1]	
	Type	Source	Type	Source	Type	Source
1	Traprock..........	Mass......	Glacial sand........	Mass......	Traprock E........	N. J.
2	.....do............	.....do....	.....do..........	.....do....	Traprock F........	Do.
3	.....do............	.....do....	.....do..........	.....do....	Rhyolite..........	Mass.
4	.....do............	.....do....	.....do..........	.....do....	Traprock..........	Do.
5	.....do............	.....do....	.....do..........	.....do....	Fly ash A........	N. J.
6	.....do............	.....do....	.....do..........	.....do....	Fly ash B........	Do.
7	.....do............	.....do....	.....do..........	.....do....	Fly ash C........	Do.
8	.....do............	.....do....	.....do..........	.....do....	Fly ash..........	Ill.
9	Rhyolite..........	.....do....	.....do..........	.....do....	Traprock E........	N. J.
10	.....do............	.....do....	.....do..........	.....do....	Traprock F........	Do.
11	.....do............	.....do....	.....do..........	.....do....	Rhyolite..........	Mass.
12	.....do............	.....do....	.....do..........	.....do....	Traprock..........	Do.
13	.....do............	.....do....	.....do..........	.....do....	Fly ash A........	N. J.
14	.....do............	.....do....	.....do..........	.....do....	Fly ash B........	Do.
15	.....do............	.....do....	.....do..........	.....do....	Fly ash C........	Do.
16	.....do............	.....do....	.....do..........	.....do....	Fly ash..........	Ill.
17	.....do............	.....do....	Potomac River sand.	D. C......	.....do...........	Do.
18	.....do............	.....do....	.....do..........	.....do....	Rhyolite..........	Mass.
19	.....do............	.....do....	.....do..........	.....do....	Mica dust........	Md.
20	Blast furnace slag..	Md.......	.....do..........	.....do....	Fly ash..........	Ill.
21	.....do............	.....do....	.....do..........	.....do....	Rhyolite..........	Mass.
22	.....do............	.....do....	.....do..........	.....do....	Mica dust........	Md.
23 [2]	Traprock..........	Mass......	Glacial sand........	Mass......	Traprock..........	Mass.
24	.....do............	.....do....	.....do..........	.....do....	Limestone........	Do.
25 [3]	.....do............	.....do....	.....do..........	.....do....	Traprock..........	Do.
26	.....do............	.....do....	.....do..........	.....do....	Limestone........	Do.
27	Traprock E........	N. J......	.....do..........	.....do....	Traprock E........	N. J.
28	.....do............	.....do....	.....do..........	.....do....	Limestone........	Mass.
29	Traprock..........	Va.......	.....do..........	.....do....	Traprock..........	Va.
30	.....do............	.....do....	.....do..........	.....do....	Limestone........	Mass.
31	Quartzite.........	Pa.......	.....do..........	.....do....	Quartzite.........	Pa.
32	.....do............	.....do....	.....do..........	.....do....	Limestone........	Mass.
33 [4]	Rhyolite..........	Mass.....	.....do..........	.....do....	Rhyolite..........	Do.
34	.....do............	.....do....	.....do..........	.....do....	Limestone........	Do.
35	Traprock E........	N. J.....	.....dr..........	.....do....	.....do...........	N. J.
36	.....do............	.....do....	.....do..........	.....do....	Traprock E........	Do.
37	.....do............	.....do....	.....do..........	.....do....	Traprock F........	Do.
38	.....do............	.....do....	.....do..........	.....do....	Silica............	Do.
39	.....do............	.....do....	.....do..........	.....do....	Fly ash A........	Do.
40	.....do............	.....do....	.....do..........	.....do....	Fly ash B........	Do.
41	.....do............	.....do....	.....do..........	.....do....	Fly ash C........	Do.
42	.....do............	.....do....	.....do..........	.....do....	Fly ash D........	Do.
43	Traprock F........	.....do....	.....do..........	.....do....	Limestone........	Do.
44	.....do............	.....do....	.....do..........	.....do....	Traprock E........	Do.
45	.....do............	.....do....	.....do..........	.....do....	Traprock F........	Do.
46	.....do............	.....do....	.....do..........	.....do....	Silica............	Do.
46x [5]	.....do............	.....do....	.....do..........	.....do....	.....do...........	Do.
47	.....do............	.....do....	.....do..........	.....do....	Fly ash A........	Do.
48	.....do............	.....do....	.....do..........	.....do....	Fly ash B........	Do.
48x [6]	.....do............	.....do....	.....do..........	.....do....	.....do...........	Do.
49	.....do............	.....do....	.....do..........	.....do....	Fly ash C........	Do.
50	.....do............	.....do....	.....do..........	.....do....	Fly ash D........	Do.
51	Traprock..........	Mass.....	.....do..........	.....do....	Traprock E........	Do.
52	.....do............	.....do....	.....do..........	.....do....	Traprock F........	Do.
53	.....do............	.....do....	.....do..........	.....do....	Fly ash A........	Do.
54	.....do............	.....do....	.....do..........	.....do....	Fly ash B........	Do.
55	.....do............	.....do....	.....do..........	.....do....	Fly ash C........	Do.
56	.....do............	.....do....	.....do..........	.....do....	Rhyolite..........	Mass.
57	.....do............	.....do....	.....do..........	.....do....	Limestone........	Do.
58	.....do............	.....do....	.....do..........	.....do....	Traprock..........	Do.
59	Rhyolite..........	.....do....	.....do..........	.....do....	Rhyolite..........	Do.
60	.....do............	.....do....	.....do..........	.....do....	Limestone........	Do.
61	.....do............	.....do....	.....do..........	.....do....	Traprock..........	Do.

[1] All fillers in mixtures 1–22, 25, and 26 sieved to pass No. 200 sieve. Mixtures 20, 21, and 22 contained about 2 parts of extra asphalt to satisfy the absorption of the slag, making the total asphalt content 7½ parts, but their effective asphalt content may be considered to be 5½ parts. Mixtures 56–61 contained 6 parts of asphalt per 100 parts of aggregate (basic mix basis) as compared to 5.5 parts for the others reported in series 3.
[2] Same as No. 4, except filler not all passing No. 200 sieve.
[3] Same as No. 4.
[4] Same as No. 11, except filler not all passing No. 200 sieve.
[5] This is a rerun of a duplicate sample of No. 46.
[6] This is a rerun of a duplicate sample of No. 48.

Table 5.—Results of the immersion-compression test on the mixtures of series 1[1]

Testing laboratory	Reported voids	Strength		
		Dry	Wet	Retained
LIMESTONE DUST MIXTURES				
	Percent	*P.s.i.*	*P.s.i.*	*Percent*
A	3.2	310	309	100
B		316	292	92
C	2.5	259	235	91
D	5.5	259	242	93
TRAPROCK DUST MIXTURES				
A	3.5	292	302	100+
B		326	306	94
C	3.1	252	229	91
D	5.7	264	246	93

[1] All mixtures contained 6½ parts of asphalt per 100 parts of aggregate, the basic mix basis. The fillers were sieved through the No. 200 sieve and only the passing portion was used. Both fillers were furnished to all the cooperators by the Massachusetts laboratory.

Table 6.—Results of the immersion-compression test on the mixtures of series 2

Testing laboratory	Filler	Source of filler	Reported voids	Strength		
				Dry	Wet	Retained
SERIES 2a.—ASPHALT CONTENT 6½ PARTS PER 100 PARTS AGGREGATE						
			Percent	*P.s.i.*	*P.s.i.*	*Percent*
D	Limestone dust	Mass	4.4	263	239	91[1]
C	Silica dust	N.J	3.2	236	203	86[2]
D	do	do	3.9	251	213	85
C	do	Md	3.9	234	212	91
D	do	do	5.5	252	204	81
D	do	do	4.5	252	198	79[3]
D	do	Mass	5.8	243	199	82
D	Fly ash	N.J	3.6	212	212	100
SERIES 2b.—ASPHALT CONTENT 5½ PARTS PER 100 PARTS AGGREGATE						
B	Limestone dust	Mass	5.9	353	283	80
D	do	do	8.2	256	225	88
B	Traprock dust	do	5.6	352	283	80
D	do	do	8.5	245	227	93
B	Silica dust	Md	6.4	367	246	67
B	do	N.J	6.3	317	278	88
D	do	Md	8.4	261	144	55
D	do	N.J	6.9	281	189	67
B	Fly ash	do	3.8	321	299	93
D	do	do	6.5	224	226	100

[1] Check test. First-run test result (see table 5) was 93 percent retained.
[2] Extending the 120° F. immersion to 7 days reduced this to 68 percent.
[3] Check test. First-run test result (see preceding line in table) was 81 percent retained.

portions, 6½ parts and 5½ parts of bitumen, would be used in the series 2 tests and that some silica dusts would be included to explore this factor. The results of the tests of series 2 indicated that the high bitumen content did, in fact, have the anticipated masking effect and it was decided that, in the mixtures for test series 3, only 5½ parts of bitumen would be used.

In series 2, as in series 1, the traprock dust performed at least as well in the laboratory test as the limestone dust. The average values of retained strength for the mixtures of series 2b were 86+ percent for traprock dust and 84 percent for limestone dust. The one sample of fly ash then available appeared to be superior to limestone dust. The retained strengths for this sample reported by laboratories B and D, series 2b, averaged 96+ percent.

Supplemental Studies

In addition to the mixtures listed in table 6, a number of additional mixtures were made and tested by variously modified procedures to develop information concerning questions that occurred to the several cooperators during the course of the main investigation. By means of such auxiliary studies, laboratory B noted that when only the portion passing the No. 200 sieve was used, both the limestone dust and the traprock dust of series 1 gave slightly better results than when the material was used as received with a small percentage retained on the No. 200 sieve. Although not particularly significant, the differences show a tendency, that has been believed to exist, for the coarser portion of so-called filler materials to fail to function as filler unless the aggregate as a whole happens to be deficient in intermediate fines. The comparisons are shown in table 7.

Effect of Extended Immersion and Reprocessing

In other auxiliary tests, laboratory C showed that silica dust, even in a rich mixture, was distinctly sensitive to extended immersion at 120° F., the retained strength after immersion dropping from 86 percent at 4 days to 68 percent at 7 days (table 6, footnote 2). These tests also showed that the same silica-dust mixture was also damaged somewhat more by immersion for 4 days at 140° F. than at 120° F., while the traprock-dust and limestone-dust mixtures apparently were not adversely affected by the higher immersion temperature. These relations are shown in table 8. It should be pointed out, however, that the mixtures used in this study of the effect of increased immersion temperature were mixtures that had been molded, cured, and tested, then reheated to 300° F., broken down or "disintegrated," remolded, and again cured for the tests at the increased immersion temperature. For this reason, the effect of temperature of immersion alone should not be

Table 7.—Comparative effect of fillers used as received and as sieved to pass the No. 200 sieve

Mixtures with—	Retained strength after 4 days immersion at 120° F.	
	Filler all passing No. 200	Filler used as received
	Percent	*Percent*
Limestone dust	92	91
Traprock dust	94	91

Table 8.—Effect of reheating and remolding previously compacted specimens of bituminous concrete

Mixtures with—	Strength					
	Dry		Wet		Retained	
	Original	Reheated, remolded	Original, immersed at 120° F.	Reheated, remolded, immersed at 140° F.	Original, immersed at 120° F.	Reheated, remolded, immersed at 140° F.
	P.s.i.	*P.s.i.*	*P.s.i.*	*P.s.i.*	*Percent*	*Percent*
Limestone dust	259	366	235	366	91	100
Traprock dust	252	363	229	328	91	90
Silica dust	236	347	203	274	86	79
Average	249	359	222	323	89	90
Average increase due to reprocessing, percent		44		45		

Mixtures with—	Strength		
	Dry	Wet	Retained
	P.s.i.	*P.s.i.*	*Percent*
Limestone dust..	582	517	89
Traprock dust..	731	655	90

considered conclusive. Of considerably more importance and value is a comparison of the unit compressive strengths shown by these mixtures before and after reheating and remolding. This comparison is also shown in table 8.

The data in table 8 show that the over-all effect of reprocessing these previously compacted specimens was to increase the average dry strength by 44 percent and the average wet strength by 45 percent. This corroborates numerous previous observations made in connection with the testing of bituminous mixtures. These observations have also indicated that where only reheating of cooled, uncompacted mixtures is necessary in order to mold test specimens, the compressive strength values are considerably higher than for specimens molded from freshly prepared mixtures that

have not been allowed to cool below a suitable molding temperature.

Tests by laboratory B on two large samples of plant mix, prepared by a paving contractor and shipped to the laboratory for test, showed the effect of reheating a cold, loose mixture and possibly other factors that could not be isolated. The compressive strengths and the results of the immersion-compression test on these mixtures are shown in table 9.

One of these two mixtures contained limestone dust and the other traprock dust. The gradings of the extracted aggregates are shown in figure 1, the small circles representing the plotting points for these two mixtures. The basic grading for the laboratory mixtures used in the cooperative study is also shown in figure 1, the points being indicated by X. From the curves, it

is apparent that the gradings of the two commercial mixtures were very similar to each other and to that of the laboratory mixtures except for slightly lower dust content in the commercial mixtures. The grade of asphalt in the commercial mixtures was not known, but is believed to have been the same as that used in the laboratory mixtures. The mixtures were, of course, molded and tested some considerable time after they were made. In spite of the probable effect of difference in age and perhaps other factors, however, the strikingly high compressive strengths of the test specimens from these two mixtures are believed to reflect primarily the effect of reheating in connection with molding. Comparison of these compressive strengths, shown in table 9, may be made with those of corresponding laboratory mixtures that were molded while hot immediately after mixing, shown in table 5.

Auxiliary tests similar to those reported in table 5 were made by laboratory A. The only change in procedure was to increase the water-bath temperature for the 4-day immersion from 120° F. to 140° F. For this set of tests, where reheating and remolding were not involved, the increased immersion temperature resulted in a decrease in wet compressive strength and

COMPOSITION OF MIXTURES

o PLANT MIXTURES			× LABORATORY MIXTURES	
SIEVE	TOTAL % PASSING		SIEVE	TOTAL % PASSING
	(a)	(b)		
$\frac{1}{2}$ IN.	100	100	$\frac{1}{2}$ IN.	100
$\frac{3}{8}$ IN.	99	100		
NO. 4	65	64	NO. 4	62
NO. 8	43	36		
			NO. 10	37
NO. 16	34	28		
NO. 30	26	22		
			NO. 40	18
NO. 50	17	14		
NO. 100	10	8		
NO. 200	6	5	NO. 200	7
ASPHALT % OF TOTAL MIXTURE	6.5	6.6	ASPHALT, PARTS PER 100 PARTS AGGREGATE	6.5
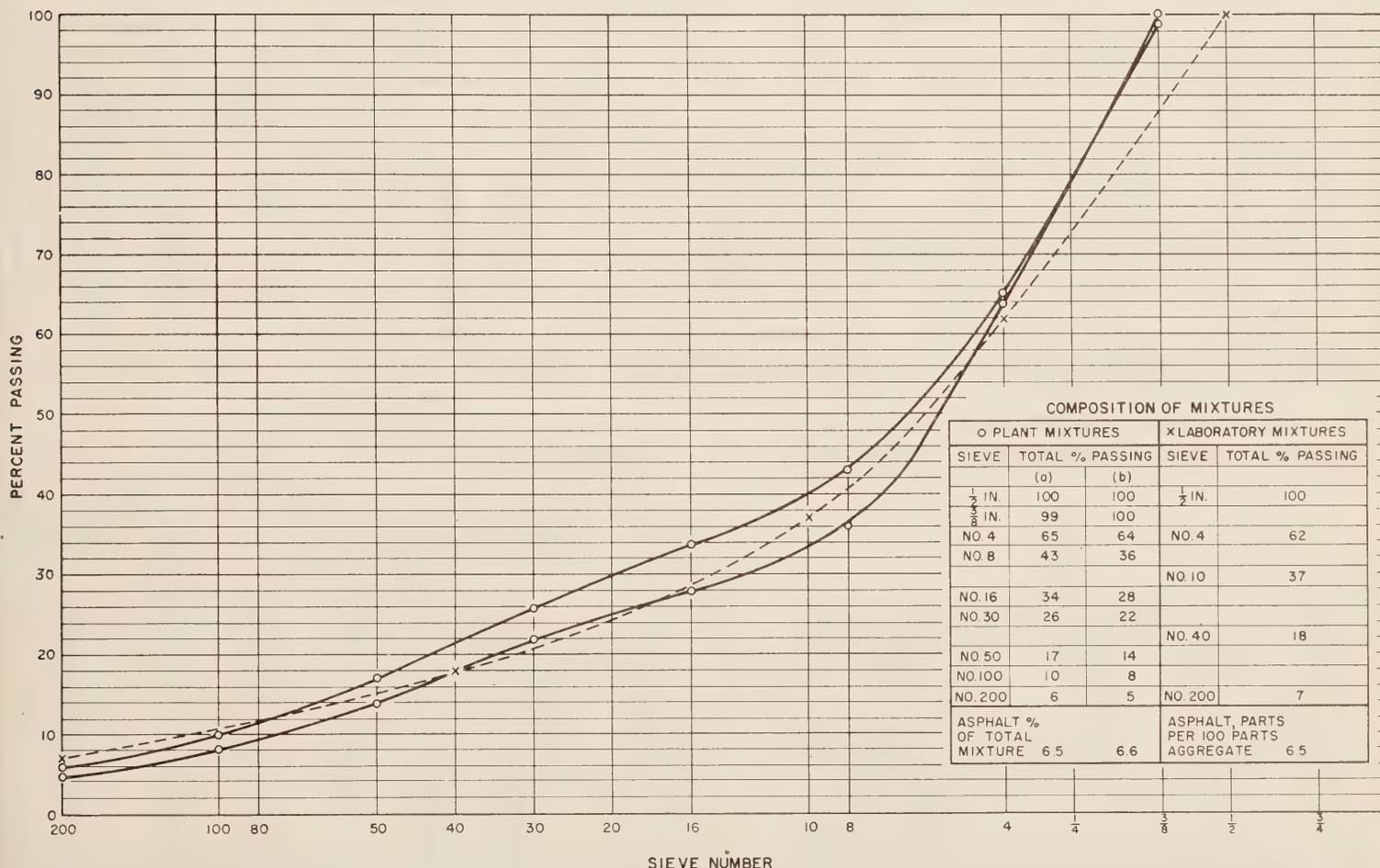
Figure 1.—Comparison of basic laboratory mixture with plant mixtures tested by laboratory B.

percentage of retained strength for both the limestone-dust and traprock-dust mixtures. This comparison is shown in table 10.

Series 3 Tests

The results of the tests by the five cooperating laboratories on the mixtures of series 3 are shown in tables 11–16. All of the mixtures of series 3, except those tested by laboratory A, contained 5½ parts of asphalt per 100 parts of aggregate. Laboratory A used 6 parts per 100. Most of the cooperating laboratories used, in addition to the regular immersion for 4 days at 120° F., one or more variations of the immersion procedure. This was done in order to develop a broader knowledge of the effect of various test conditions. The study was also expanded in series 3 to include several additional coarse aggregates, one additional sand, and several additional samples of fly ash and other fillers.

Table 11 shows the results of the tests of series 3 made by laboratory B. This table provides, in the main, a comparison between traprock dust and limestone dust as filler material, with various coarse aggregates and one type of glacial sand from Massachusetts. The two tests, involving quartzite dust in one case and rhyolite dust in the other, are auxiliary tests, as were

the tests on silica dust throughout the study. The results of these tests should not be compared directly with those of series 1 and the first part of series 2 because of the lower bitumen content of the series 3 mixtures. On the whole, the traprock dusts were indicated to be as resistant to water action in the mixtures as the limestone dust. In the standard exposure test, immersion at 120° F. for 4 days, the lowest retention for a traprock-dust mixture was 85 percent and for a limestone-dust mixture, 89 percent. The high values were 94 percent for traprock and 92 for limestone. The averages were 89+ for traprock dust and 90 for limestone dust.

It is interesting to note the small variation in percentage of retained strength for

the limestone-dust mixtures, considering the variety of coarse aggregates used. This confirms the results of other studies having to do with the application of the immersion-compression test in which it was noted that the detrimental effect of somewhat inferior coarse aggregate could be compensated by using, among other things, a high-quality filler. There is considerably more variation between the high and low values of retained strength for the traprock-dust mixtures, probably indicating some variation in the quality of the traprock dust from Massachusetts, Virginia, and New Jersey, as well as the effect of different coarse aggregates.

The high retention of strength of the quartzite-aggregate mixture with quartzite filler is interesting and somewhat surprising

Table 10.—Effect of changing the immersion temperature from 120° F. to 140° F.
(immersion-compression test, immersion period 4 days)

Mixtures with—	Strength					
	Immersion at 120° F.			Immersion at 140° F.		
	Dry	Wet	Retained	Dry	Wet	Retained
	P.s.i.	*P.s.i.*	*Percent*	*P.s.i.*	*P.s.i.*	*Percent*
Limestone dust	310	309	100	310	283	91
Traprock dust	292	302	100+	299	257	86

Table 11.—Results of series 3 immersion-compression tests [1] by laboratory B

Coarse aggregate	Fine aggregate	Filler	Air voids	Dry strength	Immersed 4 days		Immersed 14 days	
					Wet Strength	Retained strength	Wet Strength	Retained strength
			Percent	*P.s.i.*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*
Traprock (Mass.)	Glacial sand	Traprock (Mass.)	6.8	301	258	86	227	75
do	do	Limestone dust	7.0	310	281	91	267	86
do	do	Traprock (Mass.)[2]	6.1	324	275	85	230	71
do	do	Limestone dust [2]	6.3	305	280	92	274	90
Traprock E (N. J.)	do	Traprock E (N. J.)	6.5	293	276	94	242	83
do	do	Limestone dust	6.6	311	279	90	269	86
Traprock (Va.)	do	Traprock (Va.)	4.2	363	339	93	305	84
do	do	Limestone dust	5.2	330	294	89	281	85
Quartzite (Pa.)	do	Quartzite (Pa.)	4.1	375	363	97	322	86
do	do	Limestone dust	4.4	352	314	89	308	88
Rhyolite (Mass)	do	Rhyolite (Mass.)	4.3	309	163	53	55	18
do	do	Limestone dust	4.6	304	271	89	257	85

[1] All mixtures contained 5.5 parts 85–100 penetration asphalt per 100 parts of aggregate. Immersion temperature 120° F.
[2] Filler, 100 percent passing No. 200 sieve.

Table 12.—Results of series 3 immersion-compression tests [1] by laboratory C

Coarse aggregate	Fine aggregate	Filler	Air voids	Dry strength	Immersed 4 days		Immersed 7 days		Immersed 14 days	
					Wet Strength	Retained strength	Wet Strength	Retained strength	Wet Strength	Retained strength
			Percent	*P.s.i.*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*
Traprock E (N. J.)	Glacial sand (Mass.)	Limestone dust	5.5	297	235	79	228	77	217	73
do	do	Traprock E (N. J.)	5.8	290	223	77	213	73	181	62
do	do	Traprock F (N. J.)	6.0	304	236	78	211	69	196	64
do	do	Silica dust (N. J.)	5.3	277	225	81	119	43	48	17
do	do	Fly ash A (N. J.)	4.3	235	226	96	242	100+	222	94
do	do	Fly ash B (N. J.)	3.5	262	230	88	225	86	224	85
do	do	Fly ash C (N. J.)	3.7	243	246	100+	229	94	215	88
do	do	Fly ash D (N. J.)	3.2	244	222	91	235	96	225	92
Traprock F (N. J.)	do	Limestone dust	5.0	360	277	77	268	74	262	73
do	do	Traprock E (N. J.)	4.8	329	283	86	275	84	257	78
do	do	Traprock F (N. J.)	5.6	313	275	88	250	80	235	75
do	do	Silica dust (N. J.)	5.3	334	222	66	52	16	38	11
do	do	Fly ash A (N. J.)	4.0	291	277	95	272	93	267	92
do	do	Fly ash B (N. J.)	3.5	257	286	100+	277	100+	270	100+
do	do	Fly ash C (N. J.)	3.6	334	285	85	271	81	272	81
do	do	Fly ash D (N. J.)	3.2	276	278	100	283	100+	260	94

[1] All specimens tamped 25 blows on each of two layers before compressive load applied. All mixtures contained 5.5 parts 85–100 penetration asphalt per 100 parts of aggregate. Immersion temperature 120° F.

d, together with the test results on the mixtures containing New Jersey silica dust in series 2, may indicate that not all silica-dust fillers should be assumed to be unfit for use in bituminous concrete. It is important to remember, however, that final conclusions should not be drawn from laboratory results alone but should be subject to confirmation by observation of performance in field service.

Immersion Period Extended

Increasing the immersion period from 4 days to 14 days generally had a minor effect on the percentage of strength retention except in the case of the rhyolite-dust mixture, which dropped from 53 percent at 4 days to 18 percent at 14 days. In this case, the increased immersion period did not change the estimate of the value of the rhyolite filler because it would have been considered unsuitable on the basis of its 53 percent strength retention after the 4-day immersion test.

Table 12 shows the results of the tests in series 3 made by laboratory C. The groups of mixtures in the upper and lower halves of the tabulation are the same ex-

Table 13.—Results of series 3 immersion-compression tests [1] by laboratory E

Coarse aggregate	Fine aggregate	Filler	Air voids	Strength		
				Dry	Wet	Retained
			Percent	*P.s.i.*	*P.s.i.*	*Percent*
Traprock (Mass.)....	Glacial sand (Mass.).	Traprock E (N. J.)..	9.5	339	263	78
do...............	.....do.............	Traprock F (N. J.)..	9.9	355	271	76
do...............	.....do.............	Fly ash A (N. J.)....	7.2	297	278	94
do...............	.....do.............	Fly ash B (N. J.)....	7.4	315	271	86
do...............	.....do.............	Fly ash C (N. J.)....	7.5	316	273	86

[1] All mixtures contained 5.5 parts 85–100 penetration asphalt per 100 parts of aggregate. Immersion for 4 days at 120° F.

Table 14.—Partial results of series 3 immersion-compression tests [1] by laboratory A

Coarse aggregate	Fine aggregate	Filler	Air voids	Strength		
				Dry	Wet	Retained
			Percent	*P.s.i.*	*P.s.i.*	*Percent*
Traprock (Mass.)....	Glacial sand (Mass.).	Rhyolite (Mass.)....	7.0	400	253	63
do...............	.....do.............	Limestone dust......	7.0	414	288	70
do...............	.....do.............	Traprock (Mass.)....	7.7	378	277	73
Rhyolite (Mass.).....	.....do.............	Rhyolite (Mass.)....	3.9	324	232	72
do...............	.....do.............	Limestone dust......	2.9	333	361	100+
do...............	.....do.............	Traprock (Mass.)....	2.9	332	345	100+

[1] Six parts 85–100 penetration asphalt per 100 parts of aggregate used in all mixtures. All specimens in this group cured by the standard method, 24 hours at 140° F. Immersion for 14 days at 120° F.

Table 15.—Partial results of series 3 immersion-compression tests [1] by laboratory A

Test	Aggregate			Air voids	Special dry strength after additional curing 14 days at 120° F.	Immersed 4 days at 120° F. after additional curing 10 days at 120 °F.		Immersed 14 days at 120° F. after standard curing only. (24 hours at 140°F.)	
	Coarse	Fine	Filler			Wet Strength	Retained strength	Wet Strength	Retained strength
				Percent	*P.s.i.*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*
A	Traprock (Mass.)...............	Glacial sand.................	Rhyolite (Mass.).............	7.0	428	376	88	253	59
B	.....do.......................	.....do....................	Limestone dust..............	7.0	447	399	89	288	64
C	.....do.......................	.....do....................	Traprock (Mass.)............	7.7	433	393	91	277	64
	Rhyolite (Mass.)...............	.....do....................	Rhyolite (Mass.).............	3.9	427	397	93	232	54
	.....do.......................	.....do....................	Limestone dust..............	2.9	443	406	92	361	81
	.....do.......................	.....do....................	Traprock (Mass.)............	2.9	427	413	97	345	81
X	Traprock (Mass.)...............	.....do....................	Rhyolite (Mass.).............	8.5	630	533	85	357	57
Y	.....do.......................	.....do....................	Limestone dust..............	7.7	600	544	91	397	66
Z	.....do.......................	.....do....................	Traprock (Mass.)............	8.5	560	514	92	354	63

[1] Six parts 85–100 penetration asphalt per 100 parts of aggregate used in all mixtures. All specimens in this group cured by the standard method, 24 hours at 140° F. Some were then given additional curing as indicated in column heads. Tests X, Y, and Z are check runs on A, B, and C.

Table 16.—Results of series 3 immersion-compression tests [1] by laboratory D

Mixture	Coarse aggregate	Fine aggregate	Filler	Air voids	Dry strength	Immersed 4 days at 120° F.		Immersed 4 days at 140° F.		Immersed 14 days at 120° F.	
						Wet Strength	Retained strength	Wet Strength	Retained strength	Wet Strength	Retained strength
				Percent	*P.s.i.*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*	*P.s.i.*	*Percent*
1	Traprock (Mass.)......	Glacial sand (Mass.)...	Traprock E (N. J.)....	9.1	286	228	80	....		223	78
2	.....do..........	.....do............	Traprock F (N. J.)....	9.9	261	229	88	....		237	91
3	.....do..........	.....do............	Rhyolite (Mass.)......	8.7	293	70	24	48	16	42	14
4	.....do..........	.....do............	Traprock (Mass.)......	9.4	281	260	93	225	80	....	
5	.....do..........	.....do............	Fly ash A (N. J.)......	7.4	253	240	95	233	92	256	100+
6	.....do..........	.....do............	Fly ash B (N. J.)......	7.7	256	246	96	228	89	240	94
7	.....do..........	.....do............	Fly ash C (N. J.)......	7.4	255	242	95	....		218	85
8	.....do..........	.....do............	Fly ash (Ill.).........	7.3	263	252	96	251	95	228	87
9	Rhyolite (Mass.)......	.....do............	Traprock E (N. J.)....	5.5	286	238	83	231	81	....	
10	.....do..........	.....do............	Traprock F (N. J.)....	6.0	245	252	100+	255	100+	....	
11	.....do..........	.....do............	Rhyolite (Mass.)......	4.4	294	130	44	50	17	....	
12	.....do..........	.....do............	Traprock (Mass.)......	5.1	272	265	97	238	88	....	
13	.....do..........	.....do............	Fly ash A (N. J.)......	3.7	254	248	98	244	96	....	
14	.....do..........	.....do............	Fly ash B (N. J.)......	3.4	273	233	85	241	88	....	
15	.....do..........	.....do............	Fly ash C (N. J.)......	3.5	267	244	91	243	91	....	
16	.....do..........	.....do............	Fly ash (Ill.).........	3.5	261	259	99	261	100	....	
17	.....do..........	Potomac River sand....	.....do............	4.5	259	282	100+	288	100+	257	99
18	.....do..........	.....do............	Rhyolite (Mass.)......	5.5	288	116	40	43	15	50	17
19	.....do..........	.....do............	Mica dust (Md.)......	6.9	278	253	91	215	77	225	81
20	Slag (Md.)...........	.....do............	Rhyolite (Mass.)......	6.5	365	277	76	103	28	90	25
21	.....do..........	.....do............	Fly ash (Ill.).........	5.4	342	312	91	305	89	287	84
22	.....do..........	.....do............	Mica dust (Md.)......	7.1	375	314	84	279	74	252	67

[1] All mixtures except Nos. 20, 21, and 22 contained 5.5 parts 85–100 penetration asphalt. In mixtures Nos. 20, 21, and 22 about two parts per 100 of additional asphalt were used to satisfy the absorption of the slag.

Table 17.—Summary of all mixtures of series 3

Filler	Coarse aggregate	Laboratory	Voids	Dry	Wet	Retained
			Percent	P.s.i.	P.s.i.	Percent
Traprock dust (Mass.)......	Traprock (Mass.)........	B	6.8	301	258	86
do.[2]	....do	B	6.1	324	275	85
do	....do	A	7.7	433	393	[3] 91
do	....do	A	8.5	560	514	[3][4] 92
do	....do	D	9.4	281	260	93
do	Rhyolite (Mass.)........	A	2.9	427	413	[3] 97
do	....do	D	5.1	272	265	97
Traprock dust E (N. J.)....	Traprock E (N. J.)......	B	6.5	293	276	94
do	....do	C	5.8	290	223	77
do	Traprock F (N. J.)......	C	4.8	329	283	86
do	Traprock (Mass.)........	E	9.5	339	263	78
do	....do	D	9.1	286	228	80
do	Rhyolite (Mass.)........	D	5.5	286	238	83
Traprock dust F (N. J.)....	Traprock E (N. J.)......	C	6.0	304	236	78
do	Traprock F (N. J.)......	C	5.6	313	275	88
do	Traprock (Mass.)........	E	9.9	355	271	76
do	....do	D	9.9	261	229	88
do	Rhyolite (Mass.)........	D	6.0	245	252	100+
Traprock dust (Va.)........	Traprock (Va.)..........	B	4.2	363	339	93
Average percentage of retained strength, traprock dust	....					87
Limestone dust............	Traprock (Mass.)........	B	7.0	310	281	91
do.[2]	....do	B	6.3	305	280	92
do	....do	A	7.0	447	399	[3] 89
do	....do	A	7.7	600	544	[3][4] 91
do	Traprock E (N. J.)......	B	6.6	311	279	90
do	....do	C	5.5	297	235	79
do	Traprock F (N. J.)......	C	5.0	360	277	77
do	Traprock (Va.)..........	B	5.2	330	294	89
do	Rhyolite (Mass.)........	B	4.6	304	271	89
do	....do	A	2.9	443	406	[3] 92
do	Quartzite (Pa.)..........	B	4.4	352	314	89
Average percentage of retained strength, limestone dust	....					88
Fly ash A (N. J.)..........	Traprock E (N. J.)......	C	4.3	235	226	96
do	Traprock F (N. J.)......	C	4.0	291	277	95
do	Traprock (Mass.)........	E	7.2	297	278	94
do	....do	D	7.4	253	240	95
do	Rhyolite (Mass.)........	D	3.7	254	248	98
Fly ash B (N. J.)..........	Traprock E (N. J.)......	C	3.5	262	230	88
do	Traprock F (N. J.)......	C	3.5	257	286	100+
do	Traprock (Mass.)........	E	7.4	315	271	86
do	....do	D	7.7	256	246	96
do	Rhyolite (Mass.)........	D	3.4	273	233	85
Fly ash C (N. J.)..........	Traprock E (N. J.)......	C	3.7	243	246	100+
do	Traprock F (N. J.)......	C	3.6	334	285	85
do	Traprock (Mass.)........	E	7.5	316	273	86
do	....do	D	7.4	255	242	95
do	Rhyolite (Mass.)........	D	3.5	267	244	91
Fly ash D (N. J.)..........	Traprock E (N. J.)......	C	3.2	244	222	91
do	Traprock F (N. J.)......	C	3.2	276	278	100+
Fly ash (Ill.)	Traprock (Mass.)........	D	7.3	263	252	96
do	Rhyolite (Mass.)........	D	3.5	261	259	99
do	....do	D	4.5	259	282	100+
do	Slag (Md.).............	D	5.4	342	312	91
Average percentage of retained strength, fly ash	....					94
Rhyolite dust (Mass.)......	Traprock (Mass.)........	A	7.0	428	376	[3] 88
do	....do	A	8.5	630	533	[3][4] 85
do	....do	D	8.7	293	70	24
do	Rhyolite (Mass.)........	D	4.4	294	130	44
do	....do	D	5.5	288	116	40
do	....do	B	4.3	309	163	53
do	....do	A	4.1	427	397	[3] 93
do	Slag (Md.).............	D	6.5	365	277	76
Average percentage of retained strength, rhyolite dust	....					63
Quartzite dust (Pa.)........	Quartzite (Pa.)..........	B	4.1	375	363	97
Silica dust (N. J.)..........	Traprock E (N. J.)......	C	5.3	277	225	81
do	Traprock F (N. J.)......	C	5.3	334	222	66
Mica dust (Md.)...........	Rhyolite (Mass.)........	D	6.9	278	253	91
do	Slag (Md.).............	D	7.1	375	314	84

[1] Retained strengths shown for 4-day, 120° F. immersion only.
[2] Sieved to pass No. 200 sieve.
[3] Molded specimens for the dry-strength test were cured 1 day at 140° F. and 14 additional days at 120° F. Specimens for wet-strength test cured 1 day at 140° F. and 10 additional days at 120° F. before immersion.
[4] Check test of mixture reported on line next above but no explanation available for increases in dry and wet strengths.

cept for the coarse aggregate. Both coarse aggregates are traprock but they are from different sources in New Jersey. The sand used throughout was the same Massachusetts glacial material as that used in the first and second series. The traprock-dust, silica-dust, and fly-ash fillers were all New Jersey materials.

All of the mixtures containing coarse aggregate E (upper half of table 12) would be considered satisfactory on the basis of the 4-day immersion test if, as has been suggested in connection with other studi 75-percent retention is taken as the critic value. Of the mixtures containing coar aggregate F, all would be considered sat factory on the basis of the 4-day test e cept the one containing silica-dust filler.

Increasing the immersion period caus drastic additional loss of strength in both the mixtures containing silica-dust filler b did not seriously affect the other 14 mi tures.

For the standard 4-day immersion, t percentage of original dry strength retain after immersion by the four traprock-du mixtures ranged from 77 to 88, averagi 82. The retention for the eight fly-ash mi tures ranged from 85 to 100 percent, avera ing 94. The two control mixtures, contai ing limestone dust, retained 77 and 79 p cent of their dry strength after immersio Thus, the traprock-dust mixtures were i dicated by the test to be equal or superi to the limestone-dust mixtures and the fl ash mixtures were indicated to be co siderably superior to the limestone-du mixtures.

The results of the cooperative tests laboratory E are shown in table 13. He the comparison is limited to traprock du versus fly ash, with a single coarse aggr gate, but the retention values are of t same order as those previously discussed the traprock-dust mixtures being satisfa tory according to the 75-percent criteri and the fly-ash mixtures rating considerab above the suggested critical value.

In tables 14 and 15, the results of t tests by laboratory A are shown. The wo done by laboratory A in series 3 was of exploratory nature and the proportio used, 6 parts of bitumen to 100 parts aggregate, as well as various procedur that were followed, had no counterpart the work of the other laboratories. With themselves, however, the results of the tes by laboratory A show the same gener trends as those discussed in connection wi tables 11, 12, and 13. In the 4-day in mersion test, as shown in table 15, t strength retention of the limestone-dust a traprock-dust mixtures was about the sam the minor differences being slightly in fav of the traprock dust. Tests X, Y, and were check runs against A, B, and C, r spectively. The large differences betwe the dry and wet compressive strengths the two sets of tests cannot be explaine In spite of these differences, the values f percentage of retained strength are in clo agreement in the two sets of tests and, mor over, the relative values for traprock du and limestone dust are in agreement wi those of the other cooperating laboratori

The results of the series 3 tests by la oratory D are shown in table 16. A dire comparison between traprock-dust and lim stone-dust mixtures with 5½ parts of a phalt having been made in its series tests, laboratory D concentrated mainly, series 3, on obtaining a broad compariso

Table 18.—Comparison of traprock dust, fly ash, and limestone dust in series 2 and 3

Coarse aggregate	Reporting laboratory	Retained strength after immersion, 4 days, 120° F.									
		Traprock dust				Fly ash					Limestone dust
		Massachusetts	New Jersey		Virginia	New Jersey				Illinois	
			E	F		A	B	C	D		
SERIES 3 MIXTURES (5.5 parts asphalt except as noted)											
		Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent	Percent
Traprock	B	86	94	....	93	....	....	....	....	....	91
do	B	[1]85									[1]92
do	B									●	90
do	B										89
do	C		77	78	....	96	88	100+	91		79
do	C		86	88	....	95	100+	85	100		77
do	E		78	76		94	86	86			
do	A[2]	91									89
do	A[2]	92									91
do	D	93	80	88		95	96	95		96	....
Quartzite	B										89
Rhyolite	B										89
do	A[2]	97									92
do	D	97	83	100+		98	85	91		99	
do	D									100+	
Slag	D[3]									91	
SERIES 2 MIXTURES (5.5 parts asphalt only)											
Traprock	B	80	....	....		93	....	....	....		80
do	D	93	....	....		100	....	....	....		88
Averages, series 3 and 2	....	90	83	86	93	96	91	91	96	97	87

[1] Filler sieved to pass No. 200 sieve. All others tested as received.
[2] Mixtures contained 6 parts asphalt to 100 parts of aggregate (basic). Curing non-standard (see table 15).
[3] Mixture contained 2 parts of extra asphalt to satisfy absorption of slag, or a total of 7.5 parts per 100 parts of aggregate, but may be considered to contain 5.5 parts of effective asphalt.

between traprock dust and fly ash. Miscellaneous fillers, an extra coarse aggregate, and an additional sand were also included in the program for the purpose of broadening the basis for conclusions.

In the standard 4-day immersion test, the traprock-dust mixtures consistently retained 80 percent, or more, of the dry strength. The values for the six mixtures ranged from 80 to 100 percent and averaged 90 percent. Thus, all of them would be rated as satisfactory on the basis of the 75-percent criterion. Among the ten mixtures of series 3 containing fly ash, only one retained less than 90 percent of its dry strength in the 4-day, 120° F. immersion test. The range of retentions for these ten mixtures was from 85 to 100 percent and the average was 95 percent. Thus, as in the other cooperating laboratories, the fly-ash mixtures were indicated to be superior to the traprock-dust mixtures in resistance to moisture.

The rhyolite dust did not show up well in the series 3 tests by laboratory D, especially with the crushed stone aggregates. None of the rhyolite mixtures with crushed stone would be considered satisfactory on the basis of the 4-day immersion test, and the one rhyolite-dust mixture with crushed slag, while barely exceeding the suggested 75-percent criterion, was at best a borderline case.

The special tests with the immersion temperature raised to 140° F. in one case, and the immersion time increased to 14 days in the other, did not add much information concerning the relative behavior of the various fillers to that shown in the standard test, immersion for 4 days at 120° F. In general, as compared to the standard test, these special tests slightly reduced the retained strength of mixtures containing traprock dust and fly ash, but not by significant amounts. They resulted in major reductions in retained strength of the mixtures containing rhyolite filler which the standard test had already indicated to be unsatisfactory.

The tests on the natural mica dust from Maryland showed it to be about equivalent in its laboratory test behavior to the traprock dusts.

The change from glacial sand to Potomac River sand in some of the mixtures containing rhyolite coarse aggregate (see table 16) caused no significant change in the test values. Compare mixture No. 16 with No. 17, and mixture No. 11 with No. 18. Therefore, in comparing the test results on the various mixtures, the type of sand used in these tests need not be regarded as a variable factor.

Summary

All the data from tables 11–16 that were obtained from the 4-day, 120° F., immersion test have been assembled in table 17 in order to present an over-all picture for series 3. The average percentages of retained strength for the mixtures containing the various fillers indicate rhyolite to be unsatisfactory, limestone dust and traprock dust essentially equal to each other and well within the class of satisfactory materials, and fly ash superior to all the rest.

In table 18 the data from table 17, series 3 tests, pertaining to traprock dust, fly ash, and limestone dust, and those from table 6, series 2 tests, that contained 5½ parts of asphalt, have been arranged so that the range of values for retained strength obtained by the various laboratories for each of the filler samples can be more readily seen. Table 18 also indicates the degree of agreement among the cooperating laboratories on test values for individual material combinations. It is especially interesting to note that, despite some lack of agreement on the dry and wet compressive strength values and percentages of retained strength, the individual laboratories all show the same relative performance of these three filler materials. This agreement has been apparent in the preceding discussion of the individual laboratory reports, tables 11–16. Thus, the main objective of the study, to evaluate the relative laboratory performance of traprock dust, fly ash, and limestone dust as fillers in bituminous concrete, appears to have been achieved.

The related studies that were conducted by most of the cooperators, to explore the need for higher immersion temperatures or longer immersion periods in the exposure test, were of definite value in two ways. First, they indicated that generally the 4-day test at 120° F. can be relied upon to give a satisfactory separation between good and poor fillers, and second, they showed

that usually, for fillers of bad repute, the extension of the 120° F. immersion to 14 days greatly magnified the detrimental effect of the water whereas, in the case of the resistant fillers, the extended exposure caused little additional damage over that resulting from the 4-day immersion. Thus, if for any reason it should be felt that a given material showing satisfactory behavior in the 4-day test might actually not be a satisfactory filler in practice, it could be checked at the longer immersion period to clear up any doubt.

There appears to be no evidence that 4-day immersion at 140° F. would provide any more convincing information than the standard exposure for 4 days at 120° F.

Some of the other supplemental studies conducted by individual laboratories have also been useful and informative. Those of laboratory B, mentioned in connection with the discussion of table 7, showed a slight tendency for the fine portion of the mineral powder to act more effectively as a filler than when the portion retained on the No. 200 sieve was included. Those of laboratories B and C, discussed in connection with tables 8 and 9, showed in a striking way the misleading strength values that may be expected when cooled mixtures are

reheated for molding or when previously compacted mixtures are broken up, reheated, and molded.

Laboratory D made chemical analyses of four fly-ash samples, three traprock-dust samples, and one sample of rhyolite dust. The results of these analyses are given in table 19. They are presented to make available as much information as possible concerning the materials used but they are not sufficiently extensive to permit a study of

the possible effect of chemical composition on the behavior of the fillers. It may be noted that the fly-ash samples showed the lowest silica content (41 to 48 percent) and the best behavior in the test mixtures, while the traprock samples with slightly higher silica contents (50 to 52 percent) rated second in performance. The rhyolite, with the highest silica content (64 percent), showed generally unsatisfactory performance in the test mixtures.

Table 19.—Chemical analyses of fly ash, traprock, and rhyolite fillers

	Illinois	New Jersey—					Massachusetts—	
	Fly ash	Fly ash A	Fly ash B	Fly ash C	Traprock E	Traprock F	Traprock	Rhyolite
	Percent	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*	*Percent*
Ignition loss (1,000° C.)[1]	3.0	4.1	2.4	5.0	2.3	2.0	3.4	3.1
Chemical analysis:								
SiO_2	45.3	47.8	41.2	42.8	50.9	52.1	49.9	64.1
Al_2O_3	20.2	14.7	24.1	30.0	14.6	17.0	14.5	14.9
Fe_2O_3	19.4	28.6	22.1	16.1	11.3	12.0	14.0	5.3
CaO	6.3	1.5	5.6	3.7	9.7	8.6	9.3	3.6
MgO	1.1	1.2	1.3	.7	7.9	4.7	6.1	2.0
SO_3	1.8	.5	1.4	.6	.2	.1	....	....
Na_2O	1.3	.3	.7	.3	2.6	2.2	2.6	3.8
K_2O	2.0	1.9	1.2	1.4	.6	.8	.7	3.1
Total	100.4	100.6	100.0	100.6	100.1	99.5	100.5	99.9

[1] Any free carbon present is included in this percentage. In the case of the fly-ash samples, it is likely that a major part of the ignition loss represents free carbon.