Clay Mineralogy and Origin of the Huntingdon Fire Clays on Canadian Sumas Mountain, South British Columbia

Duane G. Horton
Western Washington University, dghorton@charter.net

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Date May 5, 1978

Duane G. Horton
CLAY MINERALOGY AND ORIGIN OF THE
HUNTINGDON FIRE CLAYS
ON CANADIAN SUMAS MOUNTAIN,
SOUTHWEST BRITISH COLUMBIA

A Thesis
Presented to
The Faculty of
Western Washington University

In Partial Fulfillment
Of the Requirements for the Degree
Master of Science

by
Duane G. Horton
May 1978
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Accepted in Partial Completion Of the Requirements for the Degree Master of Science

Dean of Graduate School

Advisory Committee

Chairperson
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Duane Horton
February 14, 2018
ABSTRACT

Three fire clay seams occur near the base of the Eocene Huntingdon formation at Canadian Sumas Mountain, southwest British Columbia. The three seams are separated from each other and overlain by coal partings.

The fire clays consist predominately of b-axis disordered kaolinite with minor amounts of quartz, detrital coal and x-ray amorphous iron oxides. They contain scant amounts of anatase, siderite, illite, and expandable clays. Expandable clays are concentrated in the fine clay fractions and illite is concentrated in the coarse clay fractions.

The lowermost fire clay seam contains better ordered kaolinite and fewer non-kaolinitic minerals than do the overlying two seams. Siderite shows a slight upward increase in quantity through the fire clay section. Anatase distribution is homogeneous.

All three fire clay seams are hard, compact, and non-fissile. Scanning electron microscopy shows them to be composed of small domains of fine-grained, interlocking kaolinite flakes in face-to-face contact. The lowermost seam contains ball-like aggregates of kaolinite that are not found in the overlying seams.

Field relations and mineralogic and textural features of the fire clays suggest that they were deposited as detrital mud derived from a kaolinitic saprolite which underlies the Huntingdon formation. Deposition in an Eocene flood-plain swamp is postulated.
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INTRODUCTION

Three fire clay* seams occur near the base of the Eocene Huntingdon formation at Canadian Sumas Mountain, southwest British Columbia. Because of their economic importance, previous studies have been concerned with the mining of the clays, geometry of the deposits, and various physical properties of the clays that affect their market value (Ries, 1915; Ries and Keele, 1912, 1913; Cummings and McCammon, 1952; Brady, 1961). None of the work to date includes any detailed mineralogic description of the fire clays. This study is aimed at acquiring an in-depth understanding of their mineralogy and texture and at developing a depositional model for the fire clays.

* "Fire clay" is a term used by industry to refer to refractory clays that fuse above 1520° C and do not fire to a white color (Patterson and Murray, 1975).
GEOLOGIC SETTING

The Huntingdon formation makes up part of the continental sediments deposited in southwest British Columbia and adjacent Whatcom County, Washington during Tertiary time. Fossil floras in the Huntington indicate a middle Eocene age (Fry, 1960). Rouse, however, in a personal communication with Griggs (Griggs, 1966), states that his work with the Huntingdon microfossil flora indicates an age that may be as young as Late Eocene to Oligocene.

The Huntingdon formation crops out on the southern, western, and northeastern periphery of a broad syncline that Newcomb and others (1949, in Hopkins 1966) have named the Whatcom Basin (Fig. 1). Regionally, Eocene rocks dip toward the center of the structural basin. In the north, dips are fairly shallow and only locally exceed 30°. In the south dips are steeper and, at outcrops northeast of Bellingham, reach 60° (Miller and Misch, 1963). On the east side of the basin, on the western flank of American Sumas Mountain, the Huntingdon dips 30° to 35° toward the west (Miller and Misch, 1963). The Whatcom Basin is open to the west where it enters the Strait of Georgia.

A variety of rock types are found unconformably beneath the Huntingdon formation. Northeast of Bellingham and south of the Nooksack River, about 760 meters of Huntingdon rocks (McKee, 1972; p. 100) unconformably overlie the highly folded, Late Cretaceous to Paleocene Chuckanut formation (Miller and Misch, 1963). Further northeast, between the Nooksack River and Vedder Mountain, the Huntingdon unconformably overlies metasedimentary and metavolcanic rocks of the Paleozoic Chilliwack Group; Mesozoic or Paleozoic slates, siltstones, and graywackes; and pre-late Cretaceous ultramafics.
Figure 1. Index map showing the extent of the Whatcom Basin, location of Eocene outcrops, and location of the study area (modified from Hopkins, 1966). Darkened areas are Eocene outcrops.
(Miller and Misch, 1963). In this area, on American Sumas Mountain, Miller and Misch report an exposure of about 460 meters of Huntingdon rock.

North of American Sumas Mountain, the Huntingdon formation caps the southwest end of Canadian Sumas Mountain. Here it unconformably overlies volcanics and sediments, which may represent the Chilliwack Group, and dioritic intrusives (Daly, 1912). Cummings and McCammon (1952) report 365 meters of Huntingdon formation exposed on Canadian Sumas Mountain.

Northwest of Canadian Sumas Mountain, the Huntingdon formation is covered by Pleistocene and Recent sediments. Eocene outcrops adjacent to the northwestern limit of the Whatcom Basin are assigned to the Kitsilano and Burrard formations which are roughly correlative with, but may or may not be equivalent to, the Huntingdon formation (Hopkins, 1966).
STRATIGRAPHIC DESCRIPTION OF THE HUNTINGDON FORMATION IN THE FIELD AREA

The Huntingdon formation, first described by Reginald Daly from outcrops on Canadian Sumas Mountain (Daly, 1912), consists of sandstone, siltstone, shale and a few conglomerate and coal beds.

In the field area, the Huntingdon rests uncomformably on a variable thickness of highly kaolinitized rock which is exposed in the Richmix Company's open pit mine (Figs. 2 and 3) and in roadcuts along Sumas Mountain Road (Fig. 4). The kaolinitized basement is a light-colored, massive, aphanitic rock which breaks with a blocky to conchoidal fracture. The basement rock probably represents a pre-middle Eocene episode of intense chemical weathering, a hypothesis that will be explored further during the discussion of the fire clay's origin.

Figure 5 is a generalized stratigraphic column of that part of the Huntingdon formation exposed in the study area. Figure 3 shows the lower portion of the Huntingdon formation exposed in the Richmix Pit. The section shown in Figure 3 extends from the kaolinitized basement upward to the fire clays. Underlying the fire clays are 12 to 15 meters of bedded siltstone and mudstone which vary considerably in thickness, hardness, and general outcrop appearance. Despite the diverse appearance of the beds, the mineralogy is relatively uniform and consists of quartz and kaolinite with minor amounts of illite (clay mica), hydrous iron oxides, siderite, and organic material. Most of the quartz is silt size and sub-angular. Hydrous iron oxides are present as thin stringers and small, semi-spheroidal to irregular concretionary masses. Siderite is also present as concretionary masses. Although siderite is usually not a major component of the rocks, one bed, two meters above the base of the formation, consists of about 85
Figure 2. Generalized map of the study area showing location of open pit mines and adits and the assumed extent of the fire clays and the saprolitic basement (modified from Cummings and McCammon, 1952). X denotes sample sites.
Figure 3. Kaolinitic saprolite overlain by Huntingdon formation in the Richmix Pit. Huntingdon formation section in the photo is approximately 14 meters thick.
Figure 4. Fault contact between kaolinite saprolite and illitic mudstone in roadcut along Sumas Mountain Road. Saprolite dips approximately 20° toward the viewer. Hammer is about 0.3 meters long.
SANDSTONE, CONGLOMERATE, & MUDSTONE: NOT EXPOSED IN FIELD AREA.

STRAITON PIT

NOT EXPOSED

No. 9 Pit

NOT EXPOSED

FIRE CLAY

RICHMIX PIT

KAOLINIC SAPROLITE

LOWER BOUNDARY OF HUNTINGDON FORMATION

20 METERS
percent iron oxide and siderite concretions (Fig. 6). Organic material is present in the siltstone and mudstone as small, irregular, dull to glassy blebs. One thin, extremely organic-rich bed exists about three meters above the base of the formation.

The fire clays overlie the siltstone and mudstone that is exposed in the Richmix Company Pit. To maintain stratigraphic continuity, a brief description of the fire clays is given here. A more detailed description will be given later (see RESULTS OF INVESTIGATION).

The fire clays consist of three kaolinite-rich beds which are separated from each other by thin, coal partings (Fig. 7). The beds are named, by the mine geologist, the "A", "B", and "C" seams; the "A" seam is the stratigraphically highest. The outcrop in the Richmix Pit and an outcrop immediately outside the easternmost adit (Fig. 2) are the only above ground exposures of fire clay. The fire clays are extensively exposed in underground workings. The individual seams are one to two meters thick. The fire clay is a hard, non-fissile rock that tends to break with a blocky to semi-conchoidal fracture. Its color is a light to medium shade of gray.

Overlying the "A" seam is a one meter thick coal sequence consisting of interbedded coal and flint-like clay. The rocks that immediately overlie the coal sequence are not exposed in the study area. It is necessary to skip approximately 20 meters of covered section and go to exposures in the No. 9 Pit to continue a description of the Huntingdon formation. The meager, available, drill core data indicate that the unexposed rock consist primarily of mudstone and sandstone (Cummings and McCammon, 1952).

Figure 8 shows the sequence of beds exposed in the No. 9 Pit. Approximately 10 meters of hard, buff to gray, silty mudstone are separated from
Figure 6. Photomicrograph of sample from two meters above the base of the Huntingdon formation. The rock is about 85 percent iron oxide and siderite. Crossed nicols, bar is 0.25 mm long.
Figure 7. Outcrop of fire clay in the Richmix Pit. Pack is about 0.5 meter long.
Figure 8. Sequence of beds exposed in the No. 9 Pit. Approximately 10 meters of mudstone are overlain by sandstone that is covered by Pleistocene and Recent sediments.
seven to eight meters of overlying sandstone by a thin, fissile, carbonaceous shale. The lower mudstone contains numerous iron oxide-stained fractures, infrequent slickensides, and minor quantities of detrital coal. Brady (1961), who completed a detailed study of these mudstones, reports their mineralogy to be 20 percent quartz, 9 percent siderite, 55 percent kaolinite, and 15 percent micaceous, illitic, and smectitic materials. Figure 9 is a photomicrograph and figure 10 is an x-ray diffractogram; both represent the silty mudstone.

The tan to buff sandstone is in sharp contact with the underlying carbonaceous shale. A few calcite filled fractures cut across the contact and show textures suggestive of open-space filling. The sandstone contains carbonaceous plant fossils and numerous conglomeratic lenses.

Once again, lack of exposure requires reliance on drill core data to describe the approximately 20 meters of rock overlying that seen in the No. 9 Pit. Unexposed rock includes a lower sequence of siltstone and silty mudstone; a middle sequence of sandstone; and an upper sequence of siltstone and mudstone similar to the lower sequence (Brian Stevens, personal communication, 1977).

The uppermost 90 meters of Huntingdon formation in the study area are exposed in the Straiton Pit. Figure 11 is a photograph of the lower part of the Straiton Pit section. Seven to eight meters of coarse-grained clastic rocks are overlain by about 10 meters of silty mudstone. The coarse clastics consist of sandstone and conglomerate. Both contain cross-bedding and an abundance of macroscopic carbonized plant fossils. Clasts in the conglomerate show a wide variety of lithologies including granite, diorite, quartzite, black argillite and greenschist. Hopkins (1966) says that the meager cross-
Figure 9. Photomicrograph of silty mudstone in the No. 9 Pit. Description in text, crossed nicols, bar is 0.25 mm long, M = mica, Q = quartz.
Figure 10. X-ray diffractograms of oriented, <2 µm fraction of mudstone from the No. 9 Pit. Cu Kα radiation, I = illite, K = kaolinite.
Figure 11. Sandstone and conglomerate overlain by approximately 10 meters of mudstone in the Straiton Pit.
bedding in the conglomerate indicates a northeast source area. The mudstone shown in figure 11, is a hard, non-fissile rock which breaks with a semi-conchoidal fracture. Figure 12 shows that the clay mineralogy of the mudstone is dominated by kaolinite and illite. A small peak at 4.25 Å indicates that a minor amount of quartz is present in the clay-size fraction.

Figure 13 illustrates the sequence of mudstone and siltstone in the middle part of the Straiton Pit. The mudstone ranges from greenish-gray to tan in color and from competent, ledge-forming beds to incompetent, crumbly beds. The clay mineralogy of these rocks shows a distinct change from the kaolinite- and illite-dominated mineralogy of the lower Huntingdon beds. Figure 14 is a diffractogram of a sample taken from the bed indicated by an X on figure 13. The diffractogram shows that expandable mixed-layer clays and chlorite dominate the <2 μm fraction mineralogy and that illite constitutes only a minor portion of the rock. Kaolinite, if present at all, exists in only trace amounts.

Figure 15 pictures the uppermost units in the study area. The sequence of rocks, from bottom to top, is: approximately 7 meters of blueish-green to gray mudstone and shale, 12 to 13 meters of cross-bedded sandstone, 8 meters of blueish-green to greenish-gray mudstone and shale, and a final 10 meters of conglomeratic sandstone. The sandstone contains numerous iron oxide-stained fractures, alveolar weathering cavities and a variety of primary sedimentary features that include channel structures, cut and fill structures, and cross-bedding. The mudstone and shale are variable. Generally, the greenish beds are less competent and more fissile than are the grayish ones. Most of the shale contains an abundance of organic material in a diversity of forms ranging from intricate, well preserved leaf impressions.
Figure 12. X-ray diffractograms of oriented, <2 μm fraction of mudstone shown in figure 11. Cu Kα radiation, I = illite, K = kaolinite, Q = quartz.
Figure 13. Silty mudstone and shale exposed in the Straiton Pit. Pack is about 0.5 meter long. The X denotes the bed represented by the diffractograms in figure 14.
Figure 14. X-ray diffractograms of oriented, <2 μm fraction of mudstone marked by an X on figure 13. Cu Kα radiation, C = chlorite, ML = mixed-layer clay, I = illite, K = kaolinite.
Figure 15. Uppermost mudstones and sandstones exposed in the Straiton Pit. See text for description. The X denotes the bed represented by diffractograms in figure 16.
to unrecognizable globs of coaly hash. The clay mineralogy of the upper shale and mudstone is dominated by expandable clays and chlorite (Fig. 16). Illite and kaolinite are present in only minor amounts.

Outcrops of Huntingdon formation outside of the study area and stratigraphically above the rocks in the Straiton Pit show that the upper part of the Huntingdon contains a greater percentage of coarse-grain clastics than do the lower parts.

In summary, general lithology, rapid vertical and lateral lithologic changes, and sedimentary structures imply a fluvial origin for the Huntingdon formation. Ries (1915) reached the same conclusion and said that Huntingdon sediments represent part of an Eocene Fraser River delta.

Two upward trends should be noted. The first is a general increase in coarse-grained beds toward the top of the section studied. The second trend is reflected by a change in the clay mineralogy. As the upper part of the studied section is approached, kaolinite and illite, which dominate the clays in the lower Huntingdon beds, give way to expandable clays (smectites and mixed-layer clays) and chlorite in the upper beds. The two trends may indicate a change in source rocks, a change in weathering conditions, or both.
Figure 16. X-ray diffractograms of oriented, <2 μm fraction of sample taken from the bed marked by an X in figure 15. Cu Kα radiation, C = chlorite, ML = mixed-layer clay, I = illite, K = kaolinite.
GEOMETRY OF THE FIRE CLAY DEPOSITS

Because of their economic nature, the geometry of the Huntingdon fire clay deposits has been fairly well determined (Cummings and McCammon, 1952). Figure 2 shows the extent of the fire clay deposits as deduced from outcrop and diamond drill core data. Figures 17 and 18 illustrate cross-sections through the deposits.

The eastern limit of the fire clay is in a north-south trending gully. In the gully, the Huntingdon formation unconformably overlies kaolinitized basement rock and dips uniformly, at a fairly shallow angle, to the southwest. About 100 meters west of the gully, on the north face of the Richmix Pit, some of the beds beneath the fire clays pinch out toward the east (Fig. 3). The fire clays, however, persist eastward where, in places, they directly overlie kaolinitized basement (Cummings and McCammon, 1952). About 0.4 kilometer west of the gully, drill cores and exposures in underground workings show that the basement rock rises and cuts off the fire clays along a line that is roughly parallel to the gully in the east (Cummings and McCammon, 1952). The northern and southern limits of the fire clays are not known. Drill cores indicate that the fire clays extend at least 200 meters south of the underground mine (Cummings and McCammon, 1952). Further southwest, the depositional basin deepens, but the fire clays are not present.

It appears, therefore, that the basin in which the fire clays were deposited was an irregular depression in the basement rock. This depression was about 0.4 kilometer wide, of unknown northern and southern extents, and slightly concave to the west.
Figure 17. Cross-sections A-A' and B-B' through the map shown as figure 2 (modified from Cummings and McCammon, 1952). Vertical and horizontal scales are the same. Base of cross-sections is at 100 feet elevation. Vertical lines C-C' through F-F' indicate intersections of cross-sections in figures 17 and 18.
Figure 18. Cross-sections C-C' thru F'F' through the map shown as figure 2 (modified from Cummings and McCammon, 1952). Vertical and horizontal scales are the same. Base of cross-sections is at 100 feet elevation. Vertical lines A-A' and B-B' indicate intersections of cross-sections shown in figures 17 and 18.
METHODS OF INVESTIGATION

Field Methods

Between the months of March and October, 1977, one hundred and seventy-six samples were obtained from 16 sampling sites. Sites one through ten were located in the Clayburn Company's underground workings where the fire clays are extensively exposed (Fig. 19). The remaining sites were located in three open pit mines (Fig. 2) and in roadcuts along Sumas Mountain Road. Field notes, accompanying each sample, included a brief textural and lithologic description of the sample and associated rocks and a careful notation of the exact stratigraphic and horizontal point from which the sample was obtained. This was necessary so that any vertical or lateral changes could be accurately documented. Samples were placed in sealed plastic bags for transport to the laboratory.

The fire clays were sampled at 1/3-meter stratigraphic intervals except where lithologic changes dictated denser sampling. Recent mining operations greatly aided the acquisition of fresh samples.

Laboratory Methods

A portion of each sample that was to be analysed by x-ray diffraction (XRD) was ground by hand, or by mechanical means, to pass a 0.15 mm nylon sieve. Part of the 0.15 mm powder was x-rayed in a random orientation. The remainder was ultrasonically disaggregated and the <2 μm fraction was obtained by centrifugation using speed and time data given in Jackson (1974). Oriented mounts and, in some cases, random mounts were made from the <2 μm fraction and subjected to x-ray diffraction analysis. For 12 samples, the <1 μm and <0.5 μm fractions were also obtained and x-rayed.
Figure 19. Map of the Clayburn Company underground workings (courtesy of Mr. Brian Stephens, mine manager, Clayburn Co.). Dots denote sample sites.
Normal procedure for the analysis of the clay-size fractions was to sediment a suspension of sample onto glass slides. Three slides were made per sample. One was x-rayed in an untreated state. The second slide was x-rayed after being exposed to a glycerol atmosphere according to the method described in MacEwan (1961). The third slide was x-rayed after heating to 450°C and, if the presence of chlorite was suspected, it was x-rayed a second time after heating at 575°C for 1 hour. Whenever a specimen was heat treated, appropriate care was taken to prevent rehydration. Occasionally, the above treatments were not sufficient to completely determine the clay mineralogy. For such samples treatments with K⁺ (as described by Jackson, 1974) or with HCl (as described by Brindley, 1961a) were employed. Results of all above mentioned treatments and their use in clay mineral identification are extensively discussed in Brown (1961).

All specimens were analysed with a General Electric XRD-5 diffractometer at 35 kv and 15 ma using Cu Kα radiation. A goniometer scan rate of 2° 2θ/min, a 1° beam slit, and a 0.1° receiving slit were generally used. A scanning speed of 0.4° 2θ/min and a 0.02° receiving slit were employed for polytype determinations and resolution of closely spaced reflections. Range and time constants were varied to obtain optimum diffractograms. Samples whose diffractograms (after the various treatments) were to be compared with each other were analysed at identical range and time constant settings.

Both optical and scanning electron microscopy were employed to investigate textures and to supplement XRD data. When possible, standard thin-section techniques were used. Some samples, however, crumbled when wetted. Pea-size pieces of these samples were put in small containers and covered
with a polyester resin (Fiberlay P-18). Upon hardening, standard thin-
section preparation could resume.

Scanning electron microscopy (SEM) was performed on oriented specimens
and rock chips. Oriented specimens were prepared by sedimentation of clay
suspensions. An AMR Model 1200 scanning electron microscope was used.
RESULTS OF INVESTIGATION

Lithologic and Textural Description of the Fire Clays

There are three fire clay beds, each separated from the others by coal partings. Figure 20 displays a generalized stratigraphic section of the fire clays as they appear in the underground workings.

The lowermost "C" seam is exposed only at sample sites 5 through 9 in the underground workings and in the Richmix Pit. The "C" seam is 0.5 to 1.2 meters thick and is generally thicker in the west than in the east (Cummings and McCammon, 1952). The "C" seam is a hard, non-fissile, slightly slickensided mudstone. It is moderately fractured and many of the fractures are stained orangish-brown by x-ray amorphous iron oxides. A minor amount of detrital coaly material (less than 1 percent), in the form of black, glassy blebs, is scattered throughout the clay.

Thin-sections cut from the "C" clay reveal a very fine-grained, compact, rock that is peppered with siderite concretions and sparse amounts of angular, fine sand- and silt-sized detrital quartz. The quartz grains are etched and embayed, indicating dissolution. Discrete vermicular kaolinite "books", although not common, can occasionally be seen under high power (Fig. 21). Kaolinite "books" are more abundant in the "C" seam than they are in the overlying seams. Siderite in the "C" clay assumes an interesting concretionary habit (Fig. 22). The concretions are generally less than 0.5 mm in diameter, and are almost perfectly spherical except where two or more have grown together to form linear boundaries. Commonly, detrital quartz, and less commonly pyrite and organics, are completely enclosed by the concretions. Most concretions show a pseudo-uniaxial interference figure which
Figure 20. Typical stratigraphic section of the fire clays as exposed in the underground workings (modified after Cummings and McCammon, 1952). Base of the Huntingdon lies about 15 meters below the "C" seam.
"COAL SEAM"
1.2 METERS OF COAL, HARD CLAY AND CARBONACEOUS CLAY

"A SEAM"
1.5-1.8 METERS OF FIRECLAY

0.2-0.5 METER COALY PARTING

"B SEAM"
1.5-1.8 METERS OF FIRECLAY

0.2 METER COALY PARTING

"C SEAM"
0.5-1.2 METERS OF FIRECLAY
Figure 21. Photomicrograph showing vermicular kaolinite "books" in the "C" clay. Sample is from site 5, crossed nicols, bar is 0.05 mm long, K = kaolinite.

Figure 22. Photomicrograph of siderite concretions in the "C" clay. Largest concretion is 0.5 mm across. Figure 21 is from the clay patch in upper right corner. Crossed nicols, bar is 0.5 mm long, S = siderite, Q = quartz, K = kaolinite.
indicates that they are composed of radially arranged, elongate siderite crystals. Williams, Turner, and Gilbert (1955, p. 370) mention that concretionary structures are a common habit of siderite in argillaceous rocks.

Scanning electron photomicrographs of freshly broken surface of the "C" clay reveal a very fine-grained, closely packed, compact rock. Figure 23 shows the close-packed, face-to-face arrangement of individual kaolinite flakes. The serrated edges of many of the flakes indicate partial dissolution. Figure 24 is another typical view of the "C" clay in which discrete kaolinite flakes cannot be seen. Instead, the matrix of the rock appears as a solid mass of fine-grained, interlocking particles. An interesting textural feature of the "C" clay is the tiny lumps, or balls, of kaolinite shown in figures 24 and 25. SEM photomicrographs published by Keller (Keller, 1977a, 1977b) of flint and flint-like clays show similar, although smaller and not as well formed, ball-like structures.

Overlying the "C" seam, and separating it from the "B" seam, is a 15 cm thick coal parting. The coal is dull to glassy black with a well developed blocky fracture. Many of the fractures are encrusted with orangish-brown iron oxides and, to a lesser extent, with an unidentified sulphur-like yellow material.

The "B" seam is a hard, non-fissile, medium-gray mudstone. It is generally 1.5 to 2 meters thick. At a series of underground sample sites, however, the "B" seam thins to less than 12 cm over a distance of about 14 meters (Fig. 26). This is unusual because everywhere else in the field area each fire clay seam has a relatively constant thickness. At the location where the "B" seam thins, the "C" seam makes its only underground appearance. Outcrop examination of the "B" seam discloses numerous iron oxide-
Figure 23. SEM photomicrograph of a fractured surface of the "C" clay, 5000x.

Figure 24. SEM photomicrograph of a fractured surface of the "C" clay. Note spheroidal "ball" texture, 2000x.
Figure 25. SEM photomicrograph of a fractured surface of the "C" clay. Note spheroidal "ball" texture, 2000x.
Figure 26. "A", "B", and "C" fire clay seams at sample site 5 where the "B" seam thins to approximately 12 cm. Hammer is about 0.3 meter long.
stained fractures and small, sparsely disseminated, glassy to dull fragments of detrital coal. Small fragments of coalified twigs are found in some places. Slickensides are more abundant in the "B" clay than they are in the underlying "C" clay. Slickensides are usually but not always associated with organic material. Figure 27 is a closeup of the "B" clay. Note the slickensides and fractured surfaces.

In thin-section, there are several differences between the "B" and "C" clays. The "B" clay contains more iron oxide than the "C" clay, and the oxides tend to give the whole thin-section a dirty brown appearance (Fig. 28). Detrital, angular, silt-size quartz is more plentiful in the "B" clay than it is in the "C" clay. Close examination of the quartz reveals deep embayments resulting from dissolution (Fig. 29). Small kaolinite "books", similar to those found in the "C" clay, are found but are exceedingly rare.

SEM photomicrographs (Fig. 30) show that the "B" clay consists of the same irregularly shaped, fine-grained particles that compose much of the "C" clay. Porosity may be slightly higher in the "B" clay than it is in the "C" clay, but both seams have a similar massive, welded appearance. Partial dissolution is evident from the irregular, embayed nature of some of the "B" seam kaolinite flakes. The "B" clay shows a hint of the ball-like texture found in the "C" clay, but the texture is poorly developed. In addition to kaolinite, figure 30 shows a zoned carbonate (siderite?) rhomb. No carbonate was detected in the sample by XRD. However, samples taken 10 to 12 cm above and below that of figure 30 contain siderite.

A 15 to 40 cm thick coal parting separates the "B" seam from the overlying "A" seam. In places, the parting is split into a pair of thinner partings that are separated by up to 10 cm of highly carbonaceous mudstone.
Figure 27. The "B" clay at sample site 9 showing slickensides and fractures. Knife is about 15 cm long.
Figure 28. Photomicrograph of the "B" clay. Plane polarized light, bar is 0.25 mm long, Q = quartz, O = organic clasts.

Figure 29. Photomicrograph of solution embayed quartz in the "B" clay. Crossed nicols, bar is 0.04 mm long.
Figure 30. SEM photomicrograph of a fractured surface of the "B" clay. Note zoned rhombohedral crystal of siderite(?), 5000x.

Figure 31. SEM photomicrograph of a fractured surface included in the coal parting above the "B" seam, 2000x.
The contact of the coal parting with the underlying "B" seam is gradational over a few centimeters distance. Like all other coal partings associated with the fire clays, this parting is extremely blocky and has many surfaces encrusted with iron oxides. The parting contains detrital quartz, pyrite and a variety of organic constituents including pollen and woody materials. Pyrite is found only in the carbonaceous partings. Pyrite is present as both multigranular, irregular masses and has very fine-grained blebs. Figure 31 shows the typical habit of kaolinite included in the coal parting between the "A" and "B" seams.

The uppermost fire clay seam, or "A" seam, is represented throughout the field area by 1.2 to 1.8 meters of hard, non-fissile, medium-gray mudstone. The "A" seam weathers to a light-gray color and breaks with a semi-conchoidal to blocky fracture. It contains variable but minor amounts of finely disseminated, detrital coal. The amount of detrital coal in the "A" seam increases as its upper and lower contacts with coal are approached. Slickensides and the ubiquitous iron oxide-stained fractures are abundant in the "A" clay.

Under the microscope, the "A" and "B" clays are so similar in appearance that it is difficult to distinguish one from the other. Figure 32 is a photomicrograph showing the typical appearance of the "A" clay. Figure 33 is atypical; it shows a rare occurrence of a kaolinite "book" in the "A" clay. The lack of numerous kaolinite "books" in the fire clays is in accordance with an observation made by Bates (1963), who found that hard kaolinitic clays are composed of smaller particles and contain fewer books than soft kaolinitic clays.

As with optical microscopy, scanning electron microscopy fails to show
Figure 32. Photomicrograph of the "A" clay. Crossed nicols, bar is 0.2 mm long. Small grains with high birefringence are siderite, larger detrital grains are quartz.

Figure 33. Photomicrograph of kaolinite "book" in the "A" clay. Crossed nicols, bar is 0.03 mm long, K = kaolinite, S = siderite.
any distinct differences between the "A" and "B" clays. SEM photomicrographs of the "A" clay (Fig. 34) show the tightly interlocking, compact texture that characterizes all three fire clay seams.

Keller (1976a, b, and c; 1977a and b) has published over 170 scanning electron photomicrographs of kaolins from a diversity of origins and parent materials. The Huntingdon fire clays most closely resemble Keller's sedimentary semi-flint and flint clays. Keller (1976b) gives the following textural attributes as characteristic of flint and semi-flint clays: a fine grain size, tightly compacted matrix of fine flakes and packets that are closely intergrown and interlocked, and individual particles with subhedral to anhedral morphologies. The Huntingdon fire clays are quite reminiscent of Keller's description.

The fine-grained texture of the fire clays gives a clue to the nature of the materials from which they formed. Keller (1977a) has shown SEM photomicrographs of well-formed, vermicular kaolinite books that have been mined, mechanically blended with a deflocculant, pumped several miles through a pipeline, mechanically sieved, and allowed to settle in sediment ponds. The fact that vermicular kaolinite can survive such drastic treatment suggests that it may also survive natural transportation and sedimentation. If this is true, the fine-grained nature of the fire clays may be a primary feature and not due to erosion or abrasion.

The dense interlocking textures seen in SEM photomicrographs suggest that some post-depositional crystallization has occurred. Crystallization may have taken place at the expense of primary detritus (that is, dissolution followed by precipitation) or at the expense of an amorphous aluminosilicate mud in the interstices of the detrital material.
Figure 34. SEM photomicrograph of a fractured surface of the "A" clay, 5000x.
Outcrop, thin-section, and SEM observations show a distinct lack of preferred particle orientation in the deposits. Random orientation further suggests that crystallization of an aluminosilicate mud has occurred. Alternatively, random orientation may have resulted from deposition of flocculated clays. Meade (1964) states that kaolinite tends to flocculate in edge-to-face or face-to-face arrays without developing a preferred orientation of the floccules. Small domains of kaolinite particles in face-to-face contact are readily seen in the Huntingdon fire clays.

Non-clay Mineralogy

Over seventy whole rock and more than thirty <2 μm fractions of fire clay were analysed in random orientations by x-ray diffraction. Kaolinite, illite (clay mica), quartz, anatase and siderite were identified. Ankerite was tentatively identified from two very organic-rich samples but its presence was not confirmed.

All whole rock specimens contain kaolinite and quartz as the major constituents. Their relative amounts vary quite markedly, and the variations will be discussed later (see Semi-quantitative Mineralogic Determinations). Ordinarily, quartz is a very minor constituent of the <2 μm fractions. Most quartz grains in the fire clays range in size from silt to fine sand. Figure 35 displays typical diffractograms of whole rock and <2 μm specimens; both diffractograms are from the same hand specimen.

The major anatase reflection (3.51 Å) lies on the high-angle side of the kaolinite 002 reflection (3.57 Å). Initially, the presence of anatase was confirmed by heating specimens at 550 °C for two hours. This treatment destroyed the kaolinite structure but not the anatase structure so that the
Figure 35. X-ray diffractogram of unoriented, whole rock and unoriented <2 μm fraction of fire clay. Both diffractograms are from the same hand specimen. Cu Kα radiation, K = kaolinite, Q = quartz.
anatase 3.51 Å reflection became visible. Thereafter, anatase was identified by its small 101 reflection on the high-angle side of the kaolinite 002 reflection.

Anatase is present, in trace amounts, throughout the fire clays. No other titanium-bearing mineral was identified. The widespread, although minor, occurrence of anatase in the clays is not surprising. Dimanche and Bartholomé (1976), Keller (1967), Hartman (1959), and Weaver (1976) among others, have found anatase in high-alumina deposits.

Hartman (1959), in a study of kaolinitic clays associated with bauxites, found 47 percent of the TiO₂ was anatase and 30 percent of the TiO₂ was an anatase-rutile mixture (the remaining 23 percent was rutile). Hartman (1959) found anatase to be the only TiO₂ polymorph present in Arkansas fire clays. He concluded that anatase is the common polymorph in clays whereas rutile is the common polymorph in bauxites. That anatase could not be found in thin-section, and that it was in many <2 µm fractions attests to its fine grain size in the Huntingdon clays. Hartman (1959) found the average size of anatase grains in Arkansas fire clays to be about 1 µm.

The homogenous distribution and fine grain size of the anatase coupled with its low temperature and low pressure origin (Dimanche and Bartholomé, 1976) suggest that anatase, in the fire clays, is authigenic and formed in the depositional basin by the crystallization of Ti-bearing gels (Weaver, 1976) or, less likely, from crystallization of Ti⁺⁴ in solution. It is also possible that the anatase formed by alteration of Ti-bearing minerals such as ilmenite.

Siderite was identified by its 2.79 Å reflection (32° 29). Normally, siderite was not abundant enough to produce other reflections that were not
interfered with by kaolinite reflections. Occasionally, a 1.73 Å reflection could be attributed to siderite. The existence of siderite was usually confirmed in thin-section. Some thin-sections, especially those cut from "C" seam samples, showed siderite to occur in insufficient quantities to be detected by XRD.

Siderite distribution is not homogeneous throughout the fire clays. Siderite is most abundant in organic-rich samples and attains its greatest concentration in the coal bed overlying the "A" seam. Of the three fire clay seams, the "A" seam has the greatest concentration and widest distribution of siderite. The "B" seam contains a lesser amount than does the "A" seam. Based on diffraction data alone, the occurrence of siderite in some "B" clay samples was questionable. No siderite was identified in the "C" clay by XRD, but, as shown earlier, minor quantities of siderite exist in the "C" clay as sparsely distributed concretions. XRD did not reveal siderite in any <2 μm fraction.

No unquestionable intraseam trends regarding siderite distribution were discovered. A prominent interseam trend does exist, however. Both quantity and frequency of occurrence of siderite decrease downward through the fire clay section.

Clay Mineralogy

The <2 μm fraction from 67 fire clay samples was analysed by XRD. In addition, the <1 μm and <0.5 μm fractions from 12 samples were analysed. The 12 samples represent a vertical sampling sequence through the "A" and "B" seams. Minerals identified include kaolinite, illite, inter-stratified clays, anatase and quartz. Kaolinite is, by far, the dominant mineral in all analysed samples.
The majority of diffractograms fall into one of three distinct groups. Group I is characterized by peaks at 7.13 Å and 3.56 Å representing kaolinite 001 and 002 respectively (Fig. 36). All other minerals are absent. No change occurs in group I diffractograms with glycerol or heat treatments. Sixteen fire clay samples are included in group I.

Group II (Fig. 37) has dominant peaks at 7.13 Å and 3.51 Å that, again, represent kaolinite. In addition, group II has a small but substantial broad peak between 10 Å and 11 Å. The peak indicates minor quantities of illite and mixed-layer clays. Often, the broad peak is resolved into two small, but distinct, peaks at 10 Å and somewhere near 11 Å. These peaks represent illite and interstratified illite-smectite respectively. Glyceration shifts a portion of the peak to lower 2θ angles, and the low angle background is filled in. The intensity of the 5 Å peak increases slightly with glyceration and is skewed toward high 2θ angles. Heller-Kallai and Kalman (1977) feel that such behavior is due to a physical mixture of discrete illite and randomly interstratified illite-smectite containing less than 30 percent smectite layers. An alternative explanation, given by Reynolds (Reynolds, in press), is that the illite is interstratified with 10 percent smectite. However, Reynolds has been unable to substantiate this with computer calculated XRD profiles. A third possibility is random interstratification of "vermiculitized" illite layers. Vermiculitization is a common weathering process.

After heat treatment, group II samples produce a fairly sharp 10 Å peak that is slightly larger than the 10 Å peak produced when the sample is glycerated. The heat treated 10 Å, 4.95 Å, and 3.32 Å peaks are attributed to both illite and expandable clays which have lost their interlayer water.
Figure 36. X-ray diffractogram representing group I. See text for discussion. Cu Kα radiation, oriented, <2 μm fraction, K = kaolinite.
Figure 37. X-ray diffractogram representing group II. See text for discussion. Cu Kα radiation, oriented, <2 μm fraction, K = kaolinite, I = illite, ML = mixed-layer clay.
Forty-one fire clay samples are included in group II.

A diffractogram representing group III is shown in figure 38. As with the other groups, group III is dominated by kaolinite which is unaffected by glyceration and heating. Untreated diffractograms in group III show that the low-angle background, beginning at about 8.8 2θ (10 Å), is filled in. This results from minor amounts of expandable clays in the samples. Glyceration causes slight shifts in the background, but no noticeable reflections result. The lack of 10 Å peak on glycerol-treated samples indicates little illite is present. Heating to 450° C causes collapse of expandable lattices to 10 Å. Ten fire clay samples are included in group III.

Table 1 shows the stratigraphic distribution of the sample groups. Group I, representing the purest kaolinitic clay, is the only group associated with the "C" seam. The frequency of group I samples diminishes upward. Group II samples, which contain kaolinite and both illite and interstratified clays, compose the bulk of the "A" and "B" seams. Group III samples are usually associated with coal partings.

Generally, intraseam clay mineralogy is monotonously uniform. One notable exception exists, however. At a series of underground sample sites (sites 5 through 9), the "B" seam decreases in thickness to about 12 cm. It is at this location, and at this location only, that group I samples were obtained from coal partings and from the "A" and "B" seams. With increasing distance from the pinchout, that is, as the "B" seam approaches its normal thickness, both the "A" and "B" seams take on their more characteristic group II mineralogy.
Figure 38. X-ray diffractogram representing group III. See text for discussion. Cu Ka radiation, oriented, <2 μm fraction, K = kaolinite, ML = mixed-layer clay.
Table 1. Frequency of group I, II, and III samples in each of the fire clay and coal seams.

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<th>Group I</th>
<th>Group II</th>
<th>Group III</th>
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<tr>
<td>COAL BED</td>
<td>0</td>
<td>5</td>
<td>4</td>
</tr>
<tr>
<td>&quot;A&quot; SEAM</td>
<td>2</td>
<td>24</td>
<td>0</td>
</tr>
<tr>
<td>COAL PARTING</td>
<td>2</td>
<td>4</td>
<td>4</td>
</tr>
<tr>
<td>&quot;B&quot; SEAM</td>
<td>3</td>
<td>8</td>
<td>2</td>
</tr>
<tr>
<td>COAL PARTING</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>&quot;C&quot; SEAM</td>
<td>8</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>TOTAL</td>
<td>16</td>
<td>41</td>
<td>10</td>
</tr>
</tbody>
</table>
Two extremely organic-rich samples, yielding diffractograms of the type shown in figure 39, do not fit into any of the three sample groups. The peaks at 9 Å, 4.5 Å, and 3 Å cannot be attributed to any inorganic mineral that might be expected to occur in the fire clays. Furthermore, the peaks are quite sensitive to heat. They greatly deteriorate when subjected to a 100° C glycerol atmosphere, and they are completely destroyed at 450° C. The organic-rich occurrence, heat sensitivity, and exclusion from the J.C.P.D.S. mineral file (Berry, ed., 1974) suggest that the unknown material might be a crystalline organic substance.

Twelve samples, taken at about 0.3 meter vertical intervals through the "A" and "B" seams, were fractionated into <2 μm, <1 μm, and <0.5 μm size cuts and subjected to XRD analysis. Figure 40 shows results typical of both the "A" and "B" seams. Kaolinite is dominant in all size fractions. The relative amounts of illite and interstratified clays, however, are greatly influenced by particle size. In the coarser size fraction, illite (or illite mixed with small amounts of interstratified illite-smectite with less than 10 percent smectite layers) predominates over expandable clays. In the <1 μm size fraction, the predominance of illite is diminished and interstratified clays increase in quantity as shown by the low-angle tail on the 10 Å reflection and by the high background between 8° and 6° 2θ. Finally, in the <0.5 μm fraction, illite is completely absent and expandable, interstratified clays are the only non-kaolinitic minerals present. The results shown in figure 40 agree with the findings of Hower and others (1976) who concluded, from a study of Tertiary Gulf Coast sediments, that interstratified illite-smectite is concentrated in the finer size fractions and discrete illite is concentrated in the coarser size fractions.
Figure 39. X-ray diffractogram of unidentified substance encountered in coal parting. Cu Kα radiation, oriented, <2 μm fraction, K = kaolinite, ? = unknown. See text for discussion.
Figure 40. X-ray diffractogram of <2 μm, <1 μm, and <0.5 μm fractions from the same hand specimen. Cu Kα radiation, oriented, glycerol treated, K = kaolinite, I = illite, ML = mixed-layer clay. See text for discussion.
Quantities of quartz, illite, and expandable clays relative to quantities of kaolinite, were determined from ratios of peak heights. Peak heights were measured to the nearest 0.01 inch above a smoothed general background. The kaolinite/quartz (K/Q) ratio was obtained from the height of the kaolinite 001 peak (14.4° 2θ) divided by the height of the quartz 112 peak (50.1° 2θ). The K/Q ratio was determined from randomly oriented, whole rock specimens. As the amount of quartz increases, relative to the amount of kaolinite, the ratio becomes smaller.

The illite/kaolinite (10 Å/7 Å) ratio was obtained from <2 μm oriented and glycerated specimens by dividing the height of the illite 10 Å peak (8/8° 2θ) by the height of the kaolinite 7 Å peak (12.4° 2θ). As the amount of illite increases, relative to the amount of kaolinite, the 10 Å/7 Å ratio increases.

The method used to obtain the expandable-clay/kaolinite ratio (10 Å/7 Å at 450° C - 10 Å/7 Å glycerated) is slightly more complicated. The 10 Å/7 Å ratio from an oriented, heat treated specimen should reflect the amount of illite present plus the amount of expandable clay present (collapsed to 10 Å due to heating) divided by the amount of kaolinite present. The same 10 Å/7 Å ratio from an oriented, glycerated specimen should reflect the amount of illite present divided by the amount of kaolinite present. Peaks due to illite and kaolinite remain unaffected by both glycerol and heat treatments. Therefore, any difference in the two ratios is caused by expandable clays. Furthermore, the magnitude of the difference should be proportional to the quantity of expandable clay in the sample.
Table 2. Average semi-quantitative values from each diffractogram group.

<table>
<thead>
<tr>
<th></th>
<th>Group I</th>
<th>Group II</th>
<th>Group III</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Kaolinite</strong></td>
<td>20.45</td>
<td>4.35</td>
<td>10.08</td>
</tr>
<tr>
<td><strong>Quartz</strong></td>
<td>0.03</td>
<td>0.25</td>
<td>0.19</td>
</tr>
<tr>
<td><strong>Illite</strong></td>
<td>0.05</td>
<td>1.10</td>
<td>0.12</td>
</tr>
<tr>
<td><strong>Kaolinite</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td><strong>Expandable Clay</strong></td>
<td>0.03</td>
<td>0.25</td>
<td>0.19</td>
</tr>
<tr>
<td><strong>Kaolinite</strong></td>
<td>0.05</td>
<td>1.10</td>
<td>0.12</td>
</tr>
</tbody>
</table>

Table 3. Average semi-quantitative values for each fire clay seam.

<table>
<thead>
<tr>
<th></th>
<th>Kaolinite</th>
<th>Illite</th>
<th>Expandable Clay</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Quartz</strong></td>
<td>8.76</td>
<td>0.20</td>
<td>0.07</td>
</tr>
<tr>
<td><strong>Kaolinite</strong></td>
<td>6.67</td>
<td>0.21</td>
<td>0.09</td>
</tr>
<tr>
<td><strong>Illite</strong></td>
<td>17.01</td>
<td>0.01</td>
<td>0.02</td>
</tr>
<tr>
<td><strong>Kaolinite</strong></td>
<td>10.74</td>
<td>0.21</td>
<td>0.08</td>
</tr>
<tr>
<td><strong>COAL PARTINGS</strong></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
Table 2 lists, by diffractogram group, the average value for each semi-quantitative determination. Table 3 lists, by stratigraphic position, the average value of each semi-quantitative determination. Despite a few discrepancies, the two tables correlate fairly well. For example, from table 2, group I has the highest K/Q value; from table 3, the "C" seam (composed of group I samples) has the highest K/Q value. Table 3 shows what a look at the diffractograms (figures 36, 37, and 38) has already indicated. That is, the "C" seam is composed of a more pure, kaolinitic clay than are the "A" and "B" seams. Differences between the "A" and "B" seams are less striking, but there is a slight indication that the "B" seam is poor in quartz and rich in expandable clay relative to the "A" seam. All organic-rich samples have been lumped together in table 3, and little can be said regarding the mineral content of one coal parting relative to another. Organic-rich samples as a whole, however, appear to contain quantities of quartz, relative to kaolinite, between those of the "C" seam and those of the other fire clay seams. Quantities of expandable clay and illite, relative to kaolinite, in the organic-rich samples are comparable to those of the "A" and "B" seam samples.

In addition to the interseam variations shown in table 3, two intraseam trends were noted. Generally, within invididual fire clay seams, K/Q is high directly above coal partings and gradually decreases upward. The 10 Å/7 Å values decrease from relatively high values, at the top of an individual seam, to low values near the bottom of the same seam. No significant lateral intraseam variations concerning relative amounts of minerals were noted.
Kaolinite Crystallinity Determinations

Natural kaolinite exists in a wide variety of crystalline states ranging from well-ordered kaolinite to poorly-ordered, or b-axis disordered, kaolinite. Mechanisms responsible for the disorder have been the subject of many studies. Two of the more notable ones include Bailey (1963) and Brindley and Robinson (1946).

Briefly, the three most widely accepted causes of kaolinite disorder are: 1) ± 60 rotation, normal to the c axis, of one 1:1 layer relative to adjacent 1:1 layers; 2) ± n1/3b shifts parallel to the b-axis with or without rotation; and 3) random displacements of empty octahedral sites. Recently, the rotational mechanism has fallen into disfavor. Plancon and Tchoubar (1977) have compared experimentally derived diffractograms with calculated diffractograms and concluded that random arrangement of empty octahedral sites is the main contributor to poor crystallinity in kaolinites.

The degree of disorder in a particular kaolinite is indicated in the diffraction pattern of randomly oriented samples. The intensity of reflections with k≠3n diminishes with increasing disorder, whereas reflections with k=3n remain relatively unaffected. The change in diffraction peak intensity with disorder has been the basis for quantifying kaolinite crystallinity. Johns and Murray (1959) formed a ratio to quantitatively express kaolinite crystallinity by dividing the intensity of the 02T peak (23.1°2θ) by that of the 060 peak (62.4°2θ). As disorder increases, the

* The term crystallinity refers to the degree of order-disorder in the kaolinite crystals.
intensity of the 060 remains constant, and the ratio 021/060 decreases.

Hinckley (1964) has defined a somewhat more complicated crystallinity index. Figure 41 illustrates Hinckley's method. First, a line is drawn from the trough between the 020 peak (19.9°2θ) and the 110 peak (20.4°2θ) to the background just beyond the 111 peak (21.5°2θ). Next, the intensities of the 110 and 111 peaks, above the line, are summed. Finally, the sum is divided by the intensity of the 110 peak above the general background. As kaolinite disorder increases, the Hinckley index decreases.

Both Johns and Murray's method and Hinckley's method were applied to the Huntingdon fire clays. It was necessary to measure the Hinckley index from the <2 μm fractions because the quartz 100 peak (20.9°2θ) interfered with the kaolinite peaks when coarser size fractions were used. Johns and Murray's ratio was computed from whole rock diffractograms.

In addition to the above methods, the 1/2-height width of kaolinite 001 peaks was measured on both randomly oriented, whole-rock diffractograms and on oriented, untreated, <2 μm diffractograms (Kubler, 1967, in Frey, 1970). The 1/2-height width is partially a function of crystalline order. Well-ordered kaolinites yield narrower 001 peaks than do poorly ordered kaolinites. Because peak morphology is also a function of crystallite size, domain size, peak size, and degree of particle orientation, some caution is needed when using 1/2-height width to interpret kaolinite crystallinity.

Table 4 shows the average crystallinity values for each diffractogram group. Table 5 shows the relation between crystallinity and stratigraphic position.

With one exception, all data indicate that the "C" seam (and group I)
Figure 41. Hinckley's index (from Hinckley, 1964).
Table 4. Average crystallinity determinations for each diffractogram group

<table>
<thead>
<tr>
<th></th>
<th>Group I</th>
<th>Group II</th>
<th>Group III</th>
</tr>
</thead>
<tbody>
<tr>
<td>0(^{2}1) (0\6) (0)</td>
<td>1.39</td>
<td>1.35</td>
<td>1.31</td>
</tr>
<tr>
<td>HINCKLEY'S INDEX (2)</td>
<td>0.41</td>
<td>0.27</td>
<td>0.23</td>
</tr>
<tr>
<td>1/2 HEIGHT WIDTH UNORIENTED (3)</td>
<td>0.32</td>
<td>0.38</td>
<td>0.36</td>
</tr>
<tr>
<td>1/2 HEIGHT WIDTH ORIENTED (3)</td>
<td>0.30</td>
<td>0.36</td>
<td>0.33</td>
</tr>
</tbody>
</table>

(1) Johns and Murray (1959), numbers decrease with increasing disorder.

(2) Hinckley (1964), index decreases with increasing disorder.

(3) Kubler (1967, in Frey, 1970), numbers increase with increasing disorder.
<table>
<thead>
<tr>
<th></th>
<th>$\frac{02\bar{1}}{06\bar{0}}$ (1)</th>
<th>HINCKLEY'S INDEX (2)</th>
<th>$\frac{1}{2}$ HEIGHT WIDTH UNORIENTED (3)</th>
<th>$\frac{1}{2}$ HEIGHT WIDTH ORIENTED (3)</th>
</tr>
</thead>
<tbody>
<tr>
<td>&quot;A&quot; SEAM</td>
<td>1.30</td>
<td>0.26</td>
<td>0.37</td>
<td>0.34</td>
</tr>
<tr>
<td>&quot;B&quot; SEAM</td>
<td>1.40</td>
<td>0.35</td>
<td>0.37</td>
<td>0.35</td>
</tr>
<tr>
<td>&quot;C&quot; SEAM</td>
<td>1.38</td>
<td>0.46</td>
<td>0.30</td>
<td>0.30</td>
</tr>
<tr>
<td>COAL PARTINGS</td>
<td>1.44</td>
<td>0.26</td>
<td>0.36</td>
<td>0.33</td>
</tr>
</tbody>
</table>

(1) Johns and Murray (1959), numbers decrease with increasing disorder.

(2) Hinckley (1964), index decreases with increasing disorder.

(3) Kubler (1967, in Frey, 1970), numbers increase with increasing disorder.
contains a better crystallized kaolinite than do the other fire clay seams (groups II and III). The exception, in table 5, is the $O2\overline{1}/060$ results. The average $O2\overline{1}/060$ ratios show that both the coal partings and the "B" seam are less disordered than is the "C" seam. An adequate explanation for this contradiction is not known. In the light of all other evidence in table 5, the "C" seam is considered to possess the best crystallized kaolinite.

Further inconsistencies are evident in the $O2\overline{1}/060$ results. The values show that the group III contains the kaolinite with poorest crystallinity and that the coal partings, which are dominantly group III, contain the best crystallized kaolinite. The inconsistencies encountered with the $O2\overline{1}/060$ method indicate that it is the least reliable crystallinity indicator used.

Differences in kaolinite crystallinity between the "A" seam and the "B" seam are inconsistent and much less pronounced than are differences between the "C" seam and the "A" and "B" seams. It appears that kaolinite in the "A", "B", and coal partings is comparable, and that it is more disordered than is kaolinite in the "C" seam. No intraseam variations in kaolinite crystallinity were noted.

Figure 42 shows four diffractograms which illustrate the range of kaolinite crystallinities encountered in the fire clays. Figure 42a is a diffractogram of a sample from a mudstone exposed in the No. 9 Pit which has a Hinckley index of 0.70. It is shown for comparison. Figures 42b and 42c are diffractograms of samples from the "C" seam and have Hinckley indices of 0.42 and 0.31 respectively. Figure 42d has a Hinckley index of 0.16 and represents a very disordered sample from the "A" seam.
Figure 42. Examples of kaolinite crystallinities. Crystallinity decreases from top to bottom; a. kaolinite from the No. 9 Pit, b. and c. kaolinite from the "C" seam, d. kaolinite from the "A" seam. Cu Kα radiation, unoriented.
a. Hinckley index = 0.70

b. Hinckley index = 0.42

c. Hinckley index = 0.31

d. Hinckley index = 0.16

Degrees 2θ
When compared with kaolinite from elsewhere in the world, the Huntingdon fire clays are seen to be extremely disordered. Hinckley (1964) applied his method to 144 samples of Georgia and South Carolina kaolins which were divided into two groups on the basis of physical hardness. The mean index of 72 soft kaolins was 0.91, and that of 72 hard kaolins was 0.46. Of 72 hard kaolins, Hinckley found none with an index less than 0.25. This Huntingdon study revealed 14 out of 34 samples with an index less than 0.25.

Several previous workers have shown relations between kaolinite crystallinity and particle morphology. However, few universal generalities exist. Murray and Lyons (1959) have mentioned that poorly crystallized kaolins usually lack large "books" and are composed of an agglomeration of plates that are not arranged in any precise manner. Bates (1963) agreed with Murray and Lyons when he said that coarse-grained kaolins are usually better crystallized than are fine-grained kaolins. Bates added that hard kaolins contain fewer "books" than do soft kaolins. Although a high degree of crystallinity and well-formed particle morphology are commonly characteristic of the same rock, Brindley (1961b) has cited several examples in which poorly crystallized kaolinites have yielded well-formed, hexagonal crystals.

The Huntingdon fire clays conform to the observations of Bates (1963) and Murray and Lyons (1959). The individual fire clay particles are extremely fine-grained, irregularly shaped, and stacked in an overlapping disarray. They are also quite disordered.
Summary of Data

The data indicate a homogenous mineralogy and uniform texture for each individual fire clay seam. Figure 43 illustrates all significant interseam mineralogic trends in the fire clay section.

The "C" seam generally stands out as being different from the other two seams. The "C" seam is composed of a better crystallized and slightly coarser grained kaolinite than are the "A" or "B" seams. The "C" seam contains the least amount of non-kaolinitic minerals. In addition, the "C" seam contains a ball-like micro-texture that is non-existent or, at best, poorly developed in the "A" and "B" seams.
Figure 43. Schematic stratigraphic section showing generalized trends of kaolinite crystallinity, abundance of siderite, and the ratios kaolinite/quartz, illite/kaolinite, and expandable clay/kaolinite through the three fire clay seams. Solid line = kaolinite/quartz, short dash = illite/kaolinite, alternating dash-dot = expandable clay/kaolinite, long dash = siderite abundance, dots = kaolinite crystallinity (Hinckley's index).
Increase in quantity or ratio

"A" SEAM

Siderite

Kaolinite (001)
Quartz (112)

COAL

"B" SEAM

Illite (001)
Kaolinite (001)

Hinckley's Index

COAL

"C" SEAM

Expandable clay
Kaolinite
Early Tertiary Environment

The distribution of highly aluminous sedimentary rocks is not uniform through geologic time (Keller, 1967). Notably, Pennsylvanian age and Cretaceous to Eocene age rocks contain the bulk of known bauxitic and kaolinitic deposits. Keller (1967) has mentioned that concentrations of highly aluminous sedimentary rocks coincide with periods of coal-forming environments during which marshes, swamps, and deep weathering profiles abound. Such an environment appears to have existed in the Pacific Northwest during Early Tertiary time.

Rouse (1977) has concluded, from palynological studies, that a cool, wet climate prevailed from the Arctic to south central British Columbia during the middle Paleocene. Temperate middle Paleocene conditions were followed by a warming trend in the late Paleocene which led to warm, subtropical environments throughout Eocene and earliest Oligocene time. The warming trend reached its maximum during the Late Eocene (Rouse, 1977).

Hopkins (1966), through a palynological study of Eocene rocks surrounding the Whatcom Basin, reached a similar conclusion. That is, the Eocene was characterized by warm, temperate to subtropical conditions. Hopkins further concluded that southwest British Columbia and northwest Washington comprised a low, moist, poorly drained coastal area riddled with ponds and lakes. The sporadic distribution of small Eocene deposits may reflect sedimentation in the localized basins described by Hopkins. Finally, Hopkins concluded that Eocene rainfall was approximately 130 to 160 centimeters per year.

A different line of evidence supporting warm, temperate to subtropical
Eocene environments is the nature of Early Tertiary rocks. Murray and Patterson (1975) have shown that many Eocene kaolin deposits, resulting from intense subtropical weathering, are distributed throughout the western states. In the Pacific Northwest, Eocene swamps are evident from the occurrence of coal interbeds in upper Roslyn sediments in the central Cascades (Gresens and others, 1977) and in the Huntingdon, Burrard, and Kitsilano formations in southwest British Columbia (Hopkins, 1966). Moist, warm, pre-middle Eocene conditions are suggested by iron-rich lateritic soils found below the Early Eocene Swauk formation in the northern Cascades (Gresens and others, 1977) and underlying Early Tertiary rocks at American Sumas Mountain, Whatcom County, Washington (Moen, 1969).

In summary, paleontologic and lithologic evidence indicates that early Tertiary conditions, in the Pacific Northwest, were conducive to the formation of lateritic and saprolitic deposits.

**Fire Clay Origin**

In the field area, a variable thickness of highly kaolinitic, saprolitically weathered rock underlies the Huntingdon formation. Field, thin-section, and XRD evidences all suggest that the kaolinitized saprolite developed from a hard, fine-grained, illitic mudstone. The outcrop illustrated in figure 44 shows several areas where the dark-colored, illitic mudstone was not completely kaolinitized. These areas are entirely surrounded by light-colored saprolite. The boundary between the two rock types is gradational over a distance of one to two centimeters. Thin-sections fail to show any significant textural differences across these boundaries. However, under high power, mineralogic differences are noticed.
Figure 44. Areas of relatively fresh illitic mudstone (M) surrounded by its kaolinitic saprolite (S) weathering product. Hammer is about 0.3 meter long.
The illitic mudstone contains many fractures filled with "books" of kaolinite, illite, sulphides, and calcite (Fig. 45). Similar fractures, in the saprolite, contain only kaolinite "books", siderite, and x-ray amorphous iron oxides (Fig. 46). X-ray diffractograms show an identical mineralogic difference. Figures 47 and 48 show diffractograms of samples from the illitic mudstone and the saprolite respectively. It is easily seen that the saprolite contains much less illite, relative to kaolinite, than does the mudstone. The illite, in both the mudstone and the saprolite, is a 1Md, dioctahedral variety.

Keller (1967) has described a mechanism to account for the kaolinitization of illite. The process involves incongruent dissolution of silica from illite tetrahedral sheets. Concurrently, K⁺ is taken into solution and replaced by H⁺. Thus, illite first becomes an H-rich illite and finally a kaolinite. Rainwater, with a pH of 5.5 to 6.5 (Becking and others, 1960), contributes both a supply of H⁺ ions and a medium in which the reaction can occur. The pH of rainwater can become more acidic by the addition of CO₂ and organic acids supplied from decomposing plant materials.

A kaolinitic saprolite, produced by depotassification and desilification of an illitic mudstone, is thought to be the source material from which the Huntingdon fire clays were derived. Several features of the fire clays support this hypothesis.

First, the fine-grained nature of the fire clays suggests (although not conclusively) their derivation from a fine-grained parent material.

Second, crystallinity determinations of saprolite samples are comparable to those of fire clay samples. Altshuler and others (1963) have determined that kaolinitization of muscovitic micas (compositionally
Figure 45. Photomicrograph of kaolinite and illite filled fracture in the illitic mudstone. Crossed nicols, bar is 0.05 mm long, K = kaolinite, S = siderite, I = illite.

Figure 46. Photomicrograph of kaolinite filled fracture in saprolite. Crossed nicols, bar is 0.05 mm long, K = kaolinite, S = siderite.
Figure 47. X-ray diffractogram of oriented, <2 μm fraction of illitic mudstone. Cu Kα radiation.

Figure 48. X-ray diffractogram of oriented, <2 μm fraction of kaolinitic saprolite. Cu Kα radiation.
and structurally similar to illite) yields b-axis disordered kaolinite. Eberl and Hower (1975) have found that b-axis disorder in kaolinite is favored by low temperature origin.

Third, as previously shown, the stratigraphically lower "C" seam contains less quartz, illite and interstratified clays than do the overlying two fire clay seams. This is the inverse of the soil profile expected to form on an intensely weathered, illitic mudstone. The upper, more highly kaolinitized part of the saprolite was eroded first and formed the "C" seam. This was followed by deeper erosion of less weathered rock and the deposition of less pure kaolinitic material to form the "A" and "B" seams. When the entire Huntingdon formation is considered, this trend is even more striking. The mineralogy of the lower part of the formation is almost exclusively quartz, kaolinite, and illite. As the upper part of the formation is approached, this assemblage yields to one of quartz, feldspar, mica, chlorite and expandable clays.

Thus, it appears that an illitic mudstone, weathered to produce a kaolinitic saprolite, was the parent material for the fire clays.

The fire clays are stratigraphically separated from saprolite by about 13 meters of detrital rock. The fire clays contain detrital quartz and detrital coal, and within an individual fire clay seam, there is no vertical clay-mineralogic variation suggestive of fossil soil-profiles. All this implies that the fire clays have a detrital origin and not one due directly to in-place weathering. The distance that the saprolite detritus was transported was probably short since outcrops of saprolite are found within 150 meters of the fire clay. The geometry of the fire clay deposits and their intimate association with coal suggest that a small
localized swamp or pond was the basin of deposition.

Although the saprolite is highly kaolinitic, it contains illite in quantities much greater than that found in the fire clays. Therefore, a second episode of kaolinitization is required. A swamp environment, fringed with living and decaying plant material, supplies the needed conditions to kaolinitize the illitic detritus. Plants played an indispensable role in this process.

First, they served as a mechanical filter. They kept coarse clastics from entering the basin, but still allowed the passage of fine-grained and colloidal materials. Occasionally, plant debris was swept into the basin and became incorporated in the detrital muds.

Second, living plants served as a chemical filter by extracting alkali and alkaline earth metals from interlayer positions in clay minerals. (This amounts to depotassification of illite.) Other than $K^+$ in illite, few alkali and alkaline earth ions reached the depositional basin because they were previously leached in the source area. Metals leached by living plants would be replaced by $H^+$ supplied by decaying plants. Cations taken up by plants could be removed in solution when the plants die (Keller, 1967).

Third, decaying plants produced an acidic environment in which tetrahedral silica, in illite, is slightly soluble. In addition, decaying plants created organic compounds that complexed with silica and thereby removed it from the system (Krauskopf, 1967; Keller, 1967). Thus, the same kaolinitization mechanism that operated earlier, in the source area, was at work in the depositional basin; that is, depotassification and desilification of illite to first produce an H-rich illite and then kaolinite.
Several textural features of the fire clays provide valuable clues that aid in interpreting the nature of the parent material and the conditions existing in the basin at the time of deposition. The fine-grained quality of the fire clays indicates a fine-grained parent material. This has been discussed earlier. Bates (1959) has mentioned that ball-like textures, similar to those seen in the Huntingdon clays (figures 24 and 25), are typical of allophane. Allophane is an x-ray amorphous (?) aluminosilicate material. Diagenesis may produce kaolinite from an allophane parent material. The random orientation of kaolinite particles suggests deposition from a flocculated state. Environments conducive to kaolinite flocculation are acidic and alkali and alkaline earth free (Meade, 1964). These conditions are to be expected in a swamp environment.

Some additional information is obtained by considering the environmental implications inferred from titanium-bearing and iron-bearing minerals incorporated in the fire clays.

Titanium, in the mineral anatase, occurs throughout the fire clays. A common origin for anatase is alteration of sphene, ilmenite, or some other titanium-bearing mineral (Dimanche and Bartholomé, 1976). However, the very fine-grained nature of the anatase and the fact that no other titanium-bearing minerals were encountered during the study suggests that this was not the case. Instead, the anatase is probably authigenic and formed from Ti\(^{+4}\) in solution or, more likely, from amorphous hydrous oxide gels supplied to the basin (Weaver, 1976). Authigenic anatase commonly forms from these materials in reducing, acidic sedimentary environments (Carroll, 1960; Weaver, 1976). Kessmann (1966, in Valeton, 1972) adds that low alkali concentrations favor the formation of anatase. A low alkali, reducing
acidic environment has already been proposed for the fire clays, and the
titanium mineralogy concurs.

The geochemistry and mineralogy of iron in the fire clays is more
complicated than that of titanium because several iron-bearing compounds
occur. More work is needed to accurately and thoroughly describe the
chemistry involved. However, a few general conclusions can be made.

The dotted area on figure 49 represents the Eh and pH conditions
that exist in present day peat-forming swamps (Becking and others, 1960).
A potassium concentration of 0.5 ppm is assumed (a reasonable value based
on data for swamps published in Livingston, 1963). The stable iron species
under these conditions are Fe\(^{+2}\) and Fe(OH)\(_3\). Gruner (1959) asserts that
most iron is transported, in sedimentary environments, as Fe(OH)\(_3\). Because
the parent material was weathered and eroded by surface meteoric water, it
is likely that iron was supplied to the depositional basin as Fe(OH)\(_3\) in
colloidal form. In the basin, Fe(OH)\(_3\) may have been stabilized by organic
colloids (Krauskopf, 1967; Pettijohn, 1957) or reduced to ferrous ion by
the oxidation of organic carbon.

Two crystalline iron-bearing minerals are found in the fire clays or
in the associated coal partings. Small quantities of pyrite, in coal
partings, imply that reduction of iron occurred during times of prolific
plant growth and decay or by later diagenesis.

Siderite is more abundant and widespread than is pyrite, and occurs
in clay seams as well as in coal partings. A small portion of the siderite
field in figure 49 lies within the dotted area. Post-depositional kaolinit-
tization of illite would increase the alkalinity of the environment (by
consuming H\(^+\)) so that siderite would be the favored iron compound. The
Figure 49. Diagram showing the relations among the metastable iron hydroxides and siderite and between kaolinite and illite at 25° C and 1 atm total pressure. Total activity of dissolved species = 10⁻⁰ molal. Total dissolved carbonate species = 0.01 molal. All iron data is from Garrels and Christ (1965). Illite-kaolinite boundary is for total potassium = 0.5 ppm. Free energy data for illite was taken from Helgeson (1969) and that for kaolinite from Robie and Waldbaum (1968). Dotted area encloses conditions typical for peat-forming environments as published by Becking and others (1960).
siderite-forming mechanism would be (Stashchuk, 1972):

$$4 \text{Fe(OH)}_3 + C + \text{CO}_2 + 4 \text{Fe(OH)}_2 + \text{H}_2\text{O}$$  \hspace{1cm} \text{Equation 1.}$$

followed by

$$\text{Fe(OH)}_2 + \text{CO}_2 = \text{FeCO}_3 + \text{H}_2\text{O}.$$  \hspace{1cm} \text{Equation 2.}$$

The source of carbon is organic material.

Krauskopf (1967) and Williams, Turner, and Gilbert (1954) contend that siderite concretions, similar to those shown in figure 23, form after deposition. How soon after deposition is not well understood, but Krauskopf believes they form after partial consolidation of the rock. Williams and others (1954, p. 369) simply state "Siderite spherulites in clay are apparently produced by diagenesis..." In thin-section, many of the siderite concretions are deeply stained by iron oxides. Recent weathering of siderite probably produced much of the x-ray amorphous iron oxides found in the fire clay.

Diagenesis has not only produced changes with the iron compounds, but has produced changes in the kaolinite as well. SEM photomicrographs show that the fire clays have a tightly packed, interlocking texture. This texture is not the result of sedimentation alone, nor is it entirely due to burial and compaction. Crystallization of an aluminosilicate colloidal mud (allophane?), in the intergranular interstices, or dissolution of pre-existing kaolinite followed by reprecipitation would create a hard, solid rock with interlocking or welded grains. Indeed, both crystallization of primary colloidal muds and dissolution and reprecipitation of primary kaolinite have probably occurred. It is quite feasable that the kaolinitic muds from which the fire clays formed contained amorphous aluminosilicate materials, and SEM photomicrographs clearly show ragged, serrated, kaolinite flakes which would result from dissolution.
CONCLUSIONS

1. The Huntingdon fire clays are hard, gray, non-fissile, slicken-sided mudstones. They are moderately fractured with many of the fractures stained orange-brown by x-ray amorphous iron oxides.

2. The fire clays are extremely fine-grained, compact rocks composed of small domains of interlocking kaolinite flakes in face-to-face contact.

3. Non-phyllosilicate materials present in the fire clays are predominately quartz with minor quantities of siderite, anatase, detrital coal, and amorphous iron oxides. Quartz occurs as embayed, angular, fine sand-to fine silt-size grains. Siderite occurs as submicroscopic rhombs and as spherical concretions. Anatase is present as submicroscopic grains. Iron oxides occur as fracture fillings and irregular to semi-spheroidal masses.

4. The clay mineralogy consists of kaolinite with minor amounts of illite and expandable clays. Illite is concentrated in the coarse clay size fractions and expandable clays are concentrated in the fine clay size fractions. Kaolinite dominates all size fractions.

5. Within individual fire clay seams, there is an upward increase in the quantity of quartz, relative to kaolinite, and a slight upward decrease in the quantity of illite, relative to kaolinite. There is a tendency for the siderite content to increase upward through the fire clay section. Anatase distribution is homogeneous.

6. The "C" seam contains fewer non-kaolinitic minerals than do the two overlying seams.

7. With one exception, lateral clay mineralogic variations are lacking. The one exception is a decrease in non-kaolinitic clay minerals in the "A"
where the "B" seam thins from 2 meters to 12 centimeters.

8. Kaolinite, in the fire clays, is an extremely b-axis disordered kaolinite. The only variation in kaolinite crystallinity is that the "C" seam is composed of a slightly better ordered variety than are the "A" or "B" seams.

9. The fire clays were deposited as detrital muds derived from a kaolinitic saprolite which underlies the Huntingdon formation. The saprolite is the result of intense chemical weathering of an illitic mudstone.

10. The fire clays were deposited in an Eocene flood-plain swamp that was fringed with living and decaying plants. The plants played a vital role by kaolinitizing illite incorporated in the muds supplied to the swamp.
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