X-RAY PETROLOGY OF SOME FINE GRAINED FOLIATED ROCKS

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Abstract

X-ray examination of oriented thin plates of some fine grained foliated rocks (slate and shale) shows the following:

(1) A definite relation exists between the angular spread of the reflections from the oriented platy minerals and the cleavability of the rock. The smaller the spread the better is the foliation.

(2) Greater angular spread of reflections from platy minerals results when the x-radiation is parallel to the lineation than when it is perpendicular to it. No lineation was discovered by x-ray analysis which was not easily visible in the specimen concerned.

(3) Parting of shale is explained in the same manner as foliation in schists, i.e., by parallel orientation of platy minerals.

(4) Supplementary optic examination of the quartz in one of the rocks gives a typical girdle orientation of the axes, although random orientation is indicated from its x-ray photograph.

(5) The methods employed thus far permit subdivision of the platy minerals into a kaolinite-chlorite group and a muscovite-illite group (white mica). Their relative proportions can be estimated by inspection of the chief reflection rings of each group.

(6) In the slates examined, white mica > kaolinite-chlorite where cleavability is high; kaolinite-chlorite > white mica where cleavability is low.

Introduction

Fine grained foliated rocks of the slate and shale type are not only abundant in the earth's crust, but are of great economic value. Petrologic studies of these rocks are therefore important. Until recently the petrographic microscope provided the principal means of investigation, and after a half century of service it is still the standard equipment for this purpose. However, as such studies of fine grained rocks are difficult and the results often inconclusive, the advantages of a suitable x-ray method are obvious. This paper explores a special technique and indicates ways in which further work may be undertaken. Although not the first report of its kind dealing with fine grained foliated rocks (1, 2, 3, 4), some advances in technique are outlined and conclusions offered which may prove of value.

Methods

Two types of data can be obtained from x-ray examination of mixed crystal aggregates—one compositional, the other structural. These may be obtained independently, or, as described below, in one operation. If the sample of rock is powdered in order to identify its components, obviously no structural data are obtainable from the photograph. If, however, a thin oriented plate of the rock is prepared, as first carried out by Sander (3), the photograph so obtained yields both compositional and structural data. This method, well known to metallurgists, is the one followed in the present investigation. A detailed account of the preparation of specimens and the equipment used will be found in a recent publication (5). In brief, a thin plate of the rock, ground to a thickness of .02–.04 mm., is mounted on gummed paper tape (*Sp* in Fig. 11) and placed in the path of an *x*-ray beam. This beam is reduced to a diameter of .5 mm. by a suitable collimator. The reflected radiation gives patterns on the film of the type illustrated in Figs. 1 to 8. A scanning device (Fig. 12) systematically moves the specimen to and fro in its own plane, without rotation, thus permitting the radiation to encounter a much larger number of grains than would be possible with a stationary specimen. With unfiltered radiation from a copper target the average time of exposure is 4 hours at 45 KV and 10 MA, using Eastman No-Screen *x*-ray film and Kodalk developer. This latest procedure has three advantages over that previously used: (1) thinner rock plates and faster film reduce exposure time very considerably, (2) a smaller collimator opening gives much better definition of the orientation pattern, and (3) translatory movement of the specimen (scanning) gives cleaner pictures and permits use of relatively coarse grained material.

Results of Investigation

The material selected for presentation in this paper illustrates a number of common structural and compositional features of fine grained foliated rocks. Enough accessory material was studied so that unusual characteristics could be eliminated. For this reason it was not thought necessary to include full petrographic descriptions of the rocks analyzed in Figs. 1 to 8.

All the specimens were sectioned perpendicular to the foliation. The foliation is perpendicular to each figure, with east-west orientation as shown in Fig. 13. Two features of these photographs are at once striking. One is the conspicuous, unbroken, outer ring; the other is the discontinuity of the inner rings. It can be shown by use of the Bragg equation that the unbroken outer ring is composed of reflections from rhombohedral planes of quartz. The photograph of powdered quartz in Fig. 10 provides direct evidence, showing in addition two smaller rings identifiable also in most of the rock photographs. It can likewise be shown that the discontinuous inner rings are composed of reflections from the basal planes of platy minerals such as muscovite or kaolinite.[1] The important point is that randomly oriented grains yield continuous rings, and systematically oriented grains broken rings.

A third feature, conspicuous in Figs. 1 to 4, is the spotted appearance of 1 and 3, compared with 2 and 4. The majority of the spots lie on the three quartz rings. The two photographs having this selective speckling were produced from stationary specimens, whereas Figs. 2 and 4 are from scanned specimens of the same rocks. In neither pair of photographs is there any speckling of the inner rings. The inference is that the grain size of the quartz is greater than that of the associated platy minerals. The latter do not require scanning to yield smooth rings, enough grains being encountered within the .5 mm. x-ray beam to give smooth ring segments. This conclusion is confirmed by optical study.

Figures 2 and 4 are photographs of two Vermont slates, the former a greenish-gray rock with inferior cleavage, the latter a brick-red material possessing excellent splitting properties. This contrast in cleavability is due to the varying degree of parallelism of the platy minerals in the two slates. Figure 2 shows wide ring sectors for these minerals, whereas Fig. 4 shows very narrow segments. The unoriented quartz in the two rocks plays no known role. Good foliation is indicated in Figs. 5 and 6 of a crenulated black phyllite from Nova Scotia and in Fig. 7 of a black roofing slate (locality unknown). Figure 8, on the contrary, is from a shale in which the degree of parallelism of platy minerals is relatively poor. In these and other examples studied, the excellence of the cleavage is shown by the size of ring sector in which the reflections are concentrated. By measuring the approximate angle of the ring sectors an index of cleavability could be established which might have use in commercial work. (See also Sander(3).)

The Vermont specimens (Figs. 1 to 4) show no lineation, and series of thin plates cut at various angles to each other perpendicular to the foliation give identical photographs. In contrast, the crenulated slate in Figs. 5 and 6 has marked lineation, shown both megascopically and by x-ray examination. In Fig. 6 the lineation is perpendicular to the paper, whereas in Fig. 5 it is parallel to the paper and horizontal. The spread of the inner ring reflections is noticeably greater in Fig. 6 than in Fig. 5, indicating an inferior degree of parallelism of the platy minerals where the

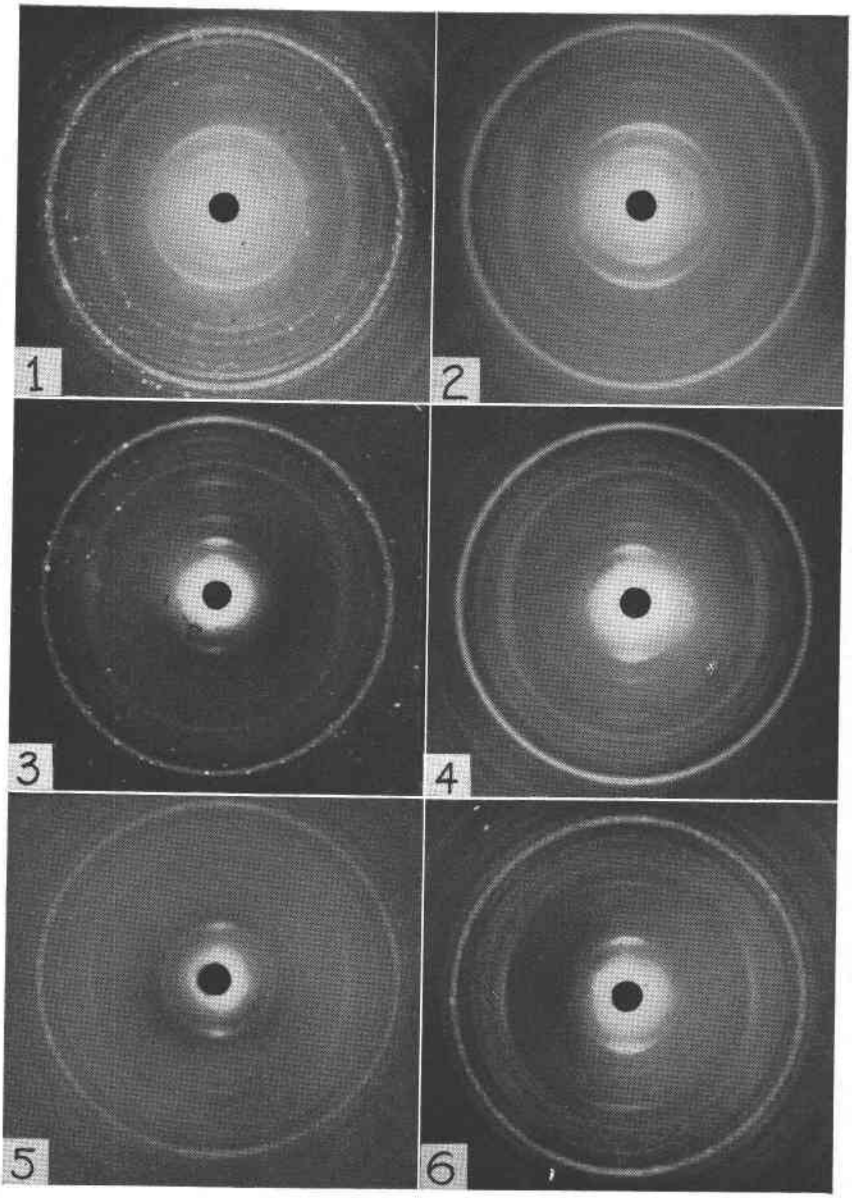
[1] Figure 9, of muscovite powder, does not show these inner rings. Apparently too few cleavage flakes in the powder are nearly enough parallel to the x-ray beam to produce the strong, low-order reflections typical of the foliated rocks. Attempts to reproduce these reflections by mixing the flakes with glass powder failed. The single conspicuous ring in Fig. 9 is referable to 006 and coincides fortuitously with the strong quartz ring of Fig. 10.

section is cut perpendicular to the lineation than in other orientations perpendicular to the lineation. This inference can be confirmed by visual examination of the rock. Sander (3) first showed this relation by *x*-ray study. It also appears, without comment, in photographs published by the author (4) in 1935.

The preceding paragraphs are concerned with structural features of the investigation alone. Additional and important information regarding the kinds and proportions of platy minerals is also obtainable. As experience has shown the desirability of obtaining basal plane reflections in work with platy minerals, the orientation of the specimens illustrated in Figs. 1 to 8 is thus highly favorable for identification purposes. Nagelschmidt (6) and others described a method for producing this orientation artificially in unconsolidated aggregates of clay minerals. In foliated rocks it is ready-made. Figure 13 shows, on the same scale as the photographs, the position of the chief basal plane reflections for kaolinite-chlorite (7.2Å) and muscovite-illite[2] (10Å). These correspond with the actual reflections in the photographs. No trace of reflections from other common platy minerals, such as serpentine and talc, was found. In the rocks producing photographs 1 to 8, routine optical examination fails to prove the presence or absence of chlorite. Rough heavy liquid fractionation and preliminary *x*-ray examination failed on the other hand to disclose any kaolinite. In consequence, therefore, reflections referable to $d_{(001)} = 7.2$Å cannot as yet be allocated. Fractionation by a supercentrifuge and thermal analysis of the products (8) will probably be necessary to decide the issue. This holds also for the white micas, muscovite and illite, which have not as yet been properly identified. The platy minerals in the rocks can be discussed, therefore, only in general terms, and only one subdivision is possible at present.

Kaolinite and chlorite on the one hand, and white mica on the other, each produce one strong basal reflection which may be used to compare relative proportions of mixtures of the two. Thus Fig. 2 shows that kaolinite-chlorite > white mica (slate with poor cleavage), whereas Fig. 4 shows that white mica > kaolinite-chlorite (slate with excellent cleavage). The crenulated black slate (Figs. 5 and 6) contains almost no kaolinite-chlorite; the roofing slate (Fig. 7) indicates a predominance of white mica; the shale (Fig. 8) has approximately equal amounts of kaolinite-chlorite and white mica. A study of the weaker reflections for each of

[2] Under illite is included the Al-poor white mica characteristic of clay sediments. As Grim (7) uses it, illite is a stop-gap term for white micaceous material which is definitely not muscovite. Much "sericite" is properly classified as illite, pending a more accurate determination.



In Figs. 1 to 8 all foliation is perpendicular to the page and is horizontal. Exposure time, about 4 hours at 10 MA, 45 KV, using Eastman No-Screen *x*-ray film and .5 mm. collimator. Thickness of specimens, .02–.04 mm. All specimens scanned except Figs. 1 and 3.

FIGS. 1 and 2. Greenish-grey slate, Poultney, Vt., collected by D. M. Larrabee. Fig. 1 is unscanned.

FIGS. 3 and 4. Red slate, Poultney, Vt., collected by D. M. Larrabee. Fig. 3 is unscanned.

FIGS. 5 and 6. Black, crenulated slate, Montague, N. S., collected by W. H. Newhouse. Fig. 5 is parallel to the foliation; Fig. 6 is perpendicular to the foliation.

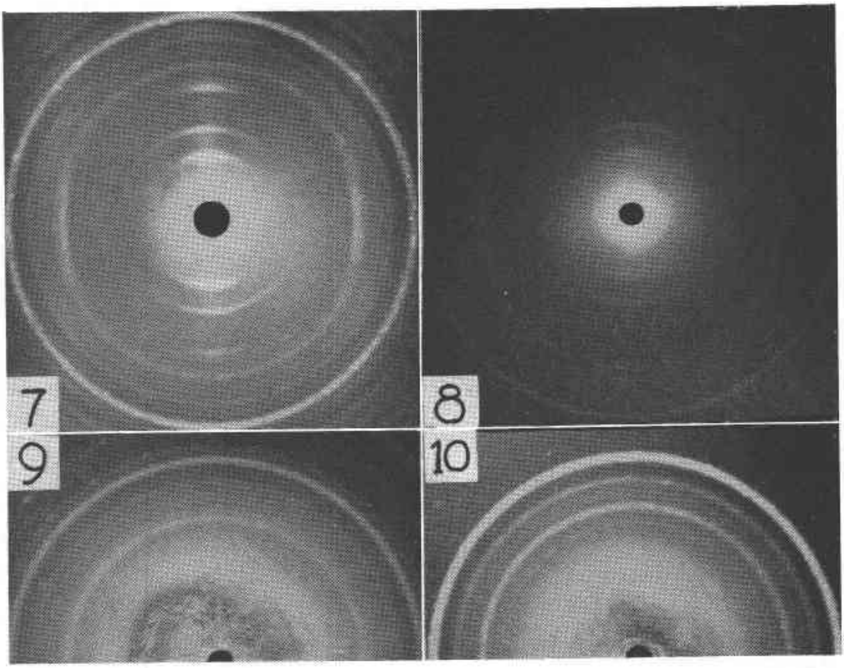


FIG. 7. Black roofing slate, locality unknown.

FIG. 8. Upper Parry shale, Kansas, collected by W. J. Mead.

FIG. 9. Powder pattern of muscovite. Note absence of reflections close to center of figure.

FIG. 10. Powder pattern of quartz.

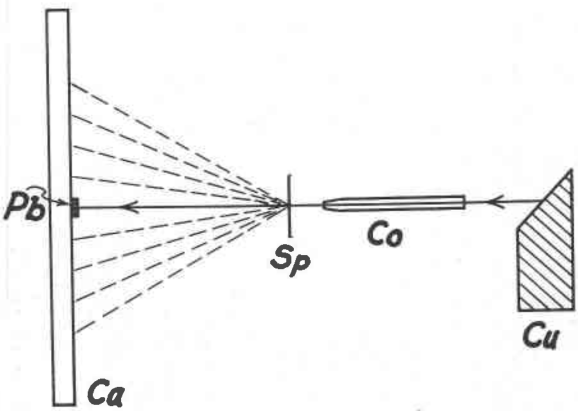


FIG. 11. Diagrammatic drawing of the essential apparatus for x-ray photography of thin slices of rocks. Cu—copper target, Co—collimator, Sp—rock specimen, Ca—cassette containing film, Pb—lead button. Broken lines indicate paths of assumed reflections from the specimen.

these minerals, in general, confirms these conclusions about relative proportions. By standardizing and refining the procedure it might be possible to determine a semi-quantitative mode for these constituents and for quartz. This has not been attempted.

Several additional features of the x-ray photographs may be noted. In Fig. 7, and less conspicuously in some others, a concentration of reflections to the right and left of the center is clearly seen. These originate from ($0kl$) planes of the vertical [001] zone (prism planes and pinacoids other than the basal) of the platy minerals. No attempt has been made to assign these reflections to particular minerals or specific planes, as they are of little significance where associated 001 reflections occur.

A second minor feature is somewhat puzzling. The basal pinacoid reflections are not always equally developed above and below the centers of the photographs. This is particularly evident in Fig. 3. In Fig. 4, a scanned photograph of the same specimen, there is approximate equality. It might be assumed that the relatively few platy grains encountered by the x-radiation to produce Fig. 3 (unscanned) were not representative of the average dimensional orientation of the slate, whereas the scanning used to produce Fig. 4 neutralized such inequalities. In Fig. 7, however, derived from a scanned slate specimen, the unequal development of reflections above and below its center is marked. More study is needed to explain this point correctly.

A third, easily overlooked, characteristic of several photographs is the lack of exact alignment of the "center of gravity" of the ring sectors above and below the center. (The position of the central black spot itself has no significance in this connection.) This non-alignment is evident in Figs. 1, 2, 3, 4, and 6 and can be checked by inspection or by use of a straight-edge. No explanation of this feature is forthcoming at present.

Discussion of Results

Although desirable for clean photographs, scanning is not essential where the investigation concerns rocks as fine grained as those described here. It is necessary only where the grain size is larger than that of normal slates and shales. Indeed, to obtain evidence of the relative grain size of quartz and associated platy minerals, a stationary specimen is required (cf. Figs. 1 and 2, 3 and 4).

Estimation of the degree of cleavability in slate (see preceding section) is obvious from a study of the hand specimen, without x-ray analysis, where the contrast is as great as that shown in Figs. 1 to 4. However, for smaller variations the x-ray method is superior if not definitive, and its possible usefulness should not be discounted.

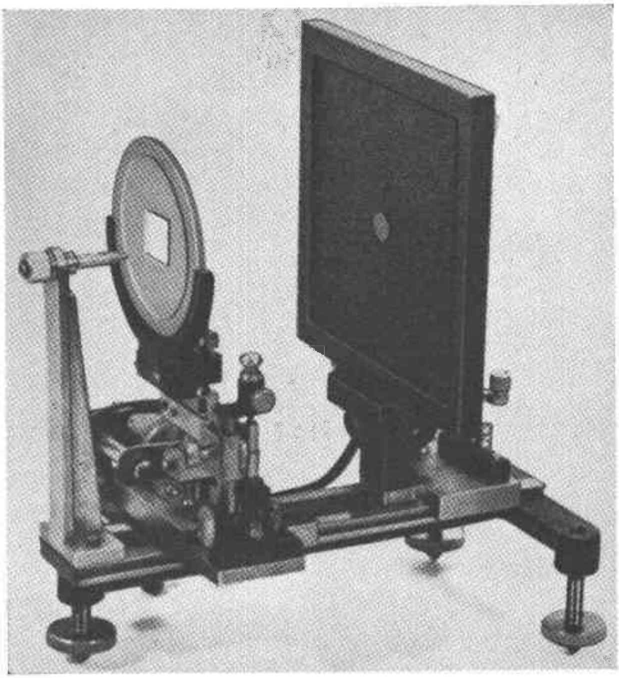
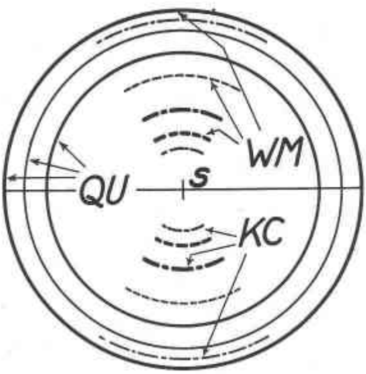


Fig. 12. Adjustable x-ray scanning camera. The mechanism beneath the specimen holder moves the specimen to and fro in parallel position across the path of the x-ray beam.



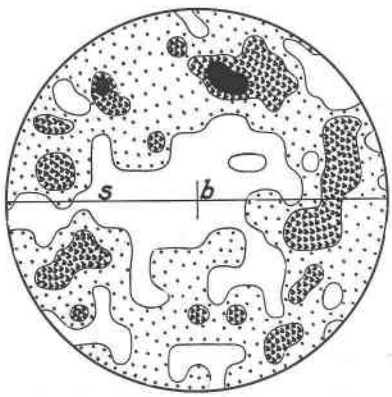


Fig. 13. Key to principal reflections observed in Figs. 1 to 8. QU—quartz, WM—muscovite and/or illite, KC—kaolinite and/or chlorite. S—trace of foliation. Intensity of reflections indicated by thickness of lines. Scale of figure same as Figs. 1 to 8.

Fig. 14. Diagram showing optic orientation of 250 quartz axes of the crenulated black slate, Montague, N. S. Figure has same orientation as Fig. 6; s—trace of foliation; b—lineation. Contours 4-3-2-1-0%.

The cleavability, or parting, of shale has been considered in some quarters as independent of parallel orientation of platy minerals, and, as petrographic proof is not easy to obtain, the problem has remained largely a matter of opinion rather than fact. Figure 8 indicates a fair degree of parallelism of the platy minerals with the parting surface of the shale. The parallelism is at least as good as that for the slate of Figs. 1 and 2, where dependence of foliation on parallelism of platy minerals is not in doubt. The evidence thus far shows that parting in shale is explained in the same way as normal foliation in other rocks.

Sander (3) was the first to study lineation by the x-ray method. Figures 5 and 6 illustrate the principle involved, as described in the preceding section. It does not seem likely, however, that the x-ray method is sensitive enough to disclose lineation not visible by inspection or optical means. The slates illustrated in Figs. 1 to 4, and in Fig. 7, without visible lineation, were studied unsuccessfully for x-ray evidence of this kind. Sander's observation, that unequal development of basal plane reflections above and below is typical of tectonites oriented as in Fig. 5 (parallel to the lineation), is not confirmed in the present study. Indeed, the opposite is true in Figs. 5 and 6. Sander assumed the inequality to be due to a "shingling" arrangement of the platy constituents, thus supporting the hypothesis that movement of material occurred in the foliation surface perpendicular to lineation. However this may be, x-ray evidence offers inconclusive support at the present time.

The random orientation of quartz[3] in these fine grained rocks, based on x-ray evidence, has already been noted. Sander (3) confirmed this by optic study, obtaining diagrams of quartz axes showing no orientation pattern. X-ray evidence, however, is not final. Optical study of the quartz in the crenulated black slate of Figs. 5 and 6 results in the orientations shown in Fig. 14, which is a common quartz pattern in schistose rocks. The failure of the x-ray method to detect quartz girdle orientation of this kind has been noted also in medium and coarse grained rocks. Unless the degree of orientation is high (as with the platy minerals in foliated rocks), optical examination is therefore necessary to complete the analysis.

The essential minerals found in these rocks—quartz, white mica,

[3] The alert reader will have noted that the main quartz ring in many of the photographs is conspicuously white above and below, and in line with, the inner discontinuous rings. This is due to the superposition of 006 muscovite reflections on the main quartz ring (cf. Figs. 9 and 10) and does not indicate quartz orientation. In support of this, it will be noted that it occurs most conspicuously in those photographs showing white mica as the predominant platy mineral.

kaolinite and/or chlorite—support the earlier optical investigations of authorities such as Behre, Dale, and Renard (9) and Berg (10). There is no confirmation of the conclusions of Anderson and Chesley (2) that kyanite, and to a lesser extent corundum, are, next to quartz, the most prominent constituents of slate. They found no muscovite or kaolinite. It is improbable that kyanite or corundum would be developed under the normal temperature-pressure conditions obtaining during slate formation. If present as detrital minerals, it is still less probable that slates from widely separated localities would all show high concentrations.

Grim and Bradley,[4] as a result of recent unpublished work on shale, find chlorite predominant over kaolinite. It is quite possible that this is true also for many slates, but, as already noted, a more thorough investigation, using the super-centrifuge and thermal analysis, will probably be required.

The relative proportions of kaolinite-chlorite and white mica have already been noted. In the slates (Figs. 1 to 7) this seems to be referable to cleavability. That is, white mica predominates where cleavage properties are well developed, and kaolinite-chlorite where cleavage is less well developed. In the single shale investigated (Fig. 8), where white mica and kaolinite-chlorite appear equally abundant, this conclusion receives neither support nor negation. Grim's study (11) of some Illinois shales shows that illite occurs instead of muscovite, and is more abundant than kaolinite. As basal plane reflections for the white micas are identical, the species of mica in the rocks under discussion is uncertain. No chemical or optical work has been undertaken in connection with this problem. Obviously, no further generalizations will be warranted until more is known of the distribution of muscovite, illite, chlorite, and kaolinite in a wide variety of fine grained foliated rocks.

Acknowledgments

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[4] Personal communication.

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