The weathering of biotite in some Aberdeenshire soils

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Summary. The weathering of biotite in some freely drained soils developed from a biotite-rich quartz gabbro near Strathdon, Aberdeenshire, has been studied. Using a combination of X-ray, chemical, differential thermal, infra-red, and optical techniques, it was found that the biotite weathers to an intergrade aluminous vermiculite–chlorite together with zones of kaolinite and gibbsite. The crystallographic axes of biotite and included kaolinite are common and parallel and it is proposed that both kaolinite and gibbsite zones crystallized epitaxially within opened out cleavage spaces of the host biotite. The development of kaolinite and gibbsite in this manner could have occurred relatively quickly and the significance of this with regard to generalized pedogenic weathering reactions of biotite is discussed.

IT has long been known that biotite releases potassium into the soil during the course of natural weathering although the associated mineralogical changes have remained obscure until fairly recently. Early studies of this problem appeared to indicate that biotite weathered towards a kaolinitic end-product. Thus, Denison *et al.* (1929), in a major study pursued mainly by chemical and optical means, came to this conclusion and Kerr (1930) described a weathered granodiorite in which biotite had changed to kaolinite via a gel-like stage. Later investigators found that biotite also weathered to hydrobiotite (Wager, 1945), vermiculite (Walker, 1949), and montmorillonite (MacEwan, 1953) and it became clear that biotite weathered to different end-products under different weathering environments. In a more recent study, Kato (1965) showed that biotite in a freely drained Japanese soil decomposed mainly to trioctahedral aluminium vermiculite and also partly to kaolinite and gibbsite. A brief preliminary account of the weathered biotites studied here (Wilson, 1966) showed them to consist of a 14 Å intergradient aluminous vermiculite-chlorite and kaolinite.

Materials and methods

Source of material. The weathered biotite used in this study was collected from three soils from the Strathdon area, Aberdeenshire. Each of these soils is freely drained and appears to be directly related to the same parent material, namely a biotite-rich quartz gabbro. This is a medium-grained holocrystalline rock whose major mineral constituents include labradorite, pyroxene, amphibole, biotite, and quartz. The pyroxene occurs as colourless, well-shaped prisms and basal plates and frequently shows various stages of alteration to amphibole. It may be surrounded by an amphibole rim or may be almost entirely replaced, the only sign of the original mineral being in the typical pyroxene shape, or in a small central core. Both augite and hypersthene are present, the former being dominant. The amphibole is usually pale green in colour, slightly or non-pleochroic, and is occasionally fibrous. It has a small or straight extinction angle and when it replaces pyroxenes it assumes a variety of orientations. X-ray powder data for the separated amphibole show a good fit for tremolite. The biotite occurs as large, deep reddish brown plates. The mafic minerals are enclosed within an unaltered subhedral mosaic of labradorite with minor amounts of interstitial quartz. Petrographically, this rock bears a close resemblance to the quartz gabbro of the Haddo House district described by Read (1935) as an uncontaminated rock showing evidence of end-stage modification and autometamorphism.

The locations of the three soils sampled are:

Soil 1.	Near the 1254-ft spot height on the west side of the minor road connecting the B973 road with Kirkton of Glen Buchat.	(NJ/372145)
Soil 2.	Approximately 1000 yards SW of Belnaboth, about 1300 ft above sea level at the head of a small stream.	(NJ/357156)
Soil 3.	On roadside just south of Tornagawn.	(NJ/343146)

The sedentary nature of Soil 1 is clear in the field since the underlying parent rock may be directly observed. The parent materials of Soils 2 and 3 could not be seen but a mineral examination indicates their uncontaminated nature. Microscopic study of the heavy and light sand fractions of the three soils showed little difference in the nature or proportions of their component minerals, although the light fraction of Soil 3 contains a little more quartz than those of Soils 1 and 2.

Methods of Investigation. Weathered biotite is very abundant in all the light, fine sand fractions (50 to 200 μ) and pure separates could be made with a magnetic separator, or simply by placing the sample on a stiff piece of paper and gently tapping so that the more rounded grains roll free leaving behind the platy biotite. The purity of each separate was always checked by examination under the binocular microscope. X-ray powder photographs were obtained using a Raymax diffraction unit (Co-$K\alpha$ radiation with an iron filter) and 9-cm precision powder cameras. Single crystal photographs were taken using a Unicam S. 35 single crystal Weissenberg goniometer (Cu-$K\alpha$ radiation with a nickel filter) the mineral having been previously correctly oriented by observing the interference figure in convergent light. Differential thermal curves were obtained in the apparatus described by Mitchell and Mackenzie (1959) using a heating rate of 10° C per minute and infra-red spectra using a Grubb-Parsons S. 4 double beam spectrometer. The cation-exchange capacities of the weathered biotites were determined by the method of Mackenzie (1951). Silica and alumina were determined colorimetrically.

Results

X-ray powder data for separated weathered biotite flakes between 50 and 200 μ in diameter from Soils 1, 2, and 3 are shown in table I, together with measurements of the fresh biotite from the parent rock of Soil 1. The results in this table are characteristic of many samples of weathered biotite taken from different parts of the three soil profiles. Several features of these powder patterns are of interest and these will be discussed in more detail.

Almost invariably a first order reflection occurs between 13·8 and 14·6 Å and at first it was considered that this spacing indicated the conversion of biotite to vermiculite, which has been shown to occur by Walker (1949). However, this interpretation was not confirmed by the behaviour of the mineral on heating or on potassium saturation. Walker (1961) showed that true vermiculite collapses to about 10 Å on heating to 300° C for several hours. Such heating of the weathered biotite, followed by immediate sealing in a glass capillary tube to prevent rehydration, induced collapse only to about 11·50 Å. Again, vermiculite derived from the weathering of biotite should collapse easily to 10 Å on potassium saturation (Weaver, 1958), but the weathered flakes failed to collapse below 13·0 Å following this treatment. These results indicated the possible presence of interlayer contaminants, in particular non-exchangeable interlayer aluminium, which has been shown to inhibit lattice contraction (Rich and Obenshain, 1955).

In order to extract interlayer aluminium the weathered flakes were treated with boiling 0·5 N NaOH for about 10 min and subsequently re-examined by X-rays. NaOH treatment brings about a significant

TABLE I. X-ray powder data for fresh and weathered biotites from soils 1, 2, and 3

Fresh Biotite		Weathered Biotite 1		Weathered Biotite 2		Weathered Biotite 3	
d	*I*	*d*	*I*	*d*	*I*	*d*	*I*
		14·5 Å	vs	13·8 Å	vs	14·4 Å	s
10·15 Å	vs	12·75	m				
		7·20	m	7·3	s	7·3	ms b
		4·85	m	4·85	s	4·75	m
4·62	w	4·65	w	4·63	m		
		4·42	w	4·40	s vb	4·48	ms bd
4·20	vw						
4·05	vw					3·88	w
3·75	m	3·60	ms	3·55	s	3·55	ms
3·35	vs			3·24	w	3·36	m
3·17	wb			3·06	w	3·14	w
2·92	wb	2·88	m	2·82	m		
2·75	s						
2·62	s	2·64	m	2·64	ms		
2·53	s			2·56	ms	2·57	m
		2·49	m				
2·44	s	2·39	m	2·39	ms	2·34	m
2·27	w						
2·23	m					2·23	w
2·20	ms						
2·05	ms			2·00	w	2·01	w
1·91	ms					1·82	vw
1·72	ms	1·68	vw	1·74	vw		
1·67	ms			1·69	vw		
1·63	vw			1·65	w		
1·61	vw						
1·54	s	1·54	ms	1·54	ms	1·54	m
		1·49	w	1·49	m	1·49	m

difference in the behaviour of the mineral on heating to 300° C and on K-saturation.

	Before NaOH treatment			After NaOH treatment		
Sample no.	1	2	3	1	2	3
Untreated	14·6	13·95	14·2	13·0	13·50	12·95
K-saturated	13·20	13·25	14·10	12·50	12·55	11·50
Heated to 300° C	11·55	11·40	11·50	10·85	10·50	10·70

Similar results were also obtained using the Na_2CO_3 method of Follett *et al.* (1965) for the dissolution of amorphous alumina and silica. The weight percentage total of Al_2O_3 extracted by this method ranged from 2·58 % to 5·40 % and clearly much of this must have been located in interlayer positions. The amount of silica extracted varied from 2·92 % to 4·67 %. Jackson (1963) has summarized recent work showing that interlayer alumination is a fairly common occurrence in expanded 2:1 layer silicates of soil clays. The interlayer positions of these silicates may be partially filled with positively charged aquo-hydroxo-aluminium ions, e.g. $Al_6(OH)_{12}(OH_2)_{12}^{6+}$, thus creating a heterogeneous intergrade between true 2:1 and 2:1:1 silicates. It seems probable that the weathered biotite has been partly converted to such an intergrade, the expanded interlayer positions being occupied by islands of hydroxo-aquo-aluminium, possibly associated with silica, and that it may be regarded, therefore, as an intergrade aluminous vermiculite-chlorite.

Table I also shows the regular occurrence of a line at about 7·3 Å, which persists on heating to 300° C indicating that it is not a second order of the 14 Å line, together with what appears to be a dioctahedral 060 line at 1·49 Å. These lines have been observed for powders and single flakes. Heating to 600° C for several hours causes them to disappear, whilst the trioctahedral 060 line associated with biotite remains unaffected. This appears to indicate the presence of a kaolinite mineral within the biotite. As to the nature of the kaolinite mineral, the slightly high spacing of the first order reflection, 7·3 Å as opposed to 7·15 Å, suggests the possibility of halloysite. The potassium acetate method of Wada (1961) was used in order to test this possibility. Flakes from Soil 3, which showed a strong line at 7·3 Å, and samples of pure kaolinite and halloysite were ground with potassium acetate. The pure minerals and the kaolinite mineral in the biotite complexed with the potassium acetate and expanded to 14 Å. When the samples were washed with water and air dried at 65 % R.H. the halloysite collapsed to 10 Å whilst the kaolinite and the kaolinite in the biotite collapsed to 7·1 and 7·3 Å respectively. It is apparent, therefore, that the behaviour of the kaolinite mineral is more like that of kaolinite than halloysite. One reason for the $00l$ spacing being at 7·3 Å may be the possible presence of a small number of interstratified 10 Å biotite units within the kaolinite crystals.

Finally, the X-ray powder data for the weathered biotites frequently show a line at about 4·8 Å. This is characteristic of gibbsite and suggests the presence of this mineral within the biotite flakes and differential thermal and infra-red results support this interpretation. The powder pattern of the biotites of Soil 3 does not contain a line at 4·8 Å indicating the absence of gibbsite in these flakes.

To recapitulate, therefore, using a combination of X-ray diffraction analysis and auxiliary chemical treatments it has been shown that biotite may weather to a complex 14 Å intergrade aluminous vermiculite-chlorite, with kaolinite and gibbsite.

X-ray single crystal results. In order to investigate the orientation relationship of the kaolinite zones in the weathered biotite a normal beam equatorial Weissenberg photograph rotated about the a-axis was taken of a weathered flake from Soil 2. All the reflections appear as broadened elongated streaks probably indicating some degree of internal disorder. The observed $00l$ and $0k0$ reflections can be attributed to a combination of weathered biotite, kaolinite, and gibbsite. To check this interpretation the flake was heated to 600° C for several hours and another zero layer photograph taken. Table II shows that those reflections attributed to kaolinite and gibbsite have disappeared. The $00l$ reflections of the heated flake now fall into a series of orders indicating collapse of the weathered biotite along the c-axis to 9·3 Å. Along the b-axis the 2·23 Å 040 kaolinite reflection vanishes on heating, whilst the 1·54 Å reflection, previously very diffuse and so obscuring a separate dioctahedral reflection at 1·49 Å, becomes much more sharply defined. These results appear to indicate that the crystallographic axes of the biotite and included kaolinite zones are common and parallel. The gibbsite is probably similarly oriented.

TABLE II. X-ray single crystal data for weathered biotite flake from Soil 2

d_{0k0}	I	Mineral	d_{00l}	I	Mineral
Weathered biotite flake					
4·60	s	Biotite	4·80	m	Gibbsite
2·23	w	Kaolinite	3·50	s	Kaolinite
1·54	vs (broad)	Biotite	3·02	w	Biotite
			2·17	ms	Biotite
			2·05	ms	Biotite
			1·44	w	
			1·30	w	
Weathered biotite flake heated at 600° C					
4·67	ms	Biotite	3·08	s	Biotite
1·54	s	Biotite	2·33	vw	Biotite
			1·86	vw	Biotite
			1·54	w	Biotite
			1·32	w	Biotite

Differential thermal curves for weathered biotites collected from the upper and lower parts of the soil profiles are shown in fig. 1. It is clear that the curves of the biotites from Soils 1 and 2 are similar—with the two most prominent endothermic reactions at about 275° C and between 480° C and 500° C. In addition there are weak endothermic effects at about 600° C and 800° C in some of the samples. Almost certainly the gibbsite present in the flakes is responsible for the reactions at about 275° C whilst the major endotherm is probably due to the dehydroxylation of kaolinite. The biotites from Soil 3 produce a different thermal curve. Here the gibbsite reaction is absent, thus confirming the X-ray data. The flakes from the upper part of soil profile 3 show a series of reactions characteristic of kaolinite and here again it may be noted that the powder pattern of this material showed a strong line at 7·3 Å. Biotite from the lower part of this soil profile lacks the high temperature exothermic reaction of kaolinite but shows a large endothermic peak at 815° C. The reason for this is unknown.

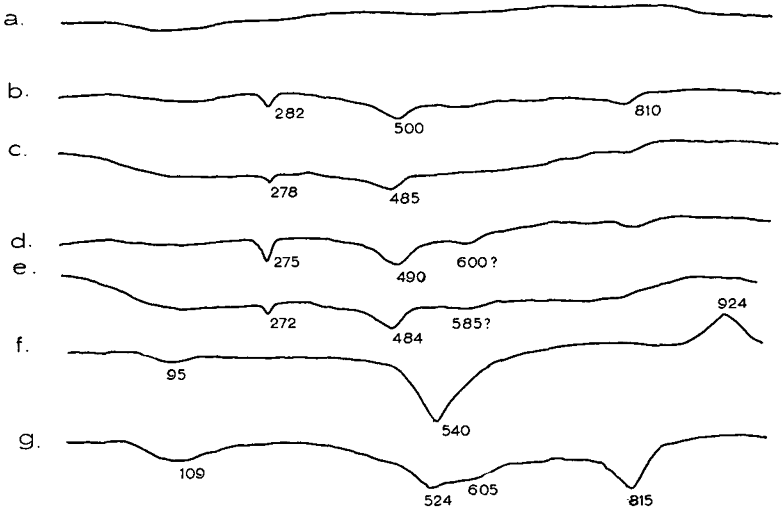

FIG. 1. Differential thermal analysis curves for: *a*, unweathered biotite; *b* to *g*, weathered biotites, from: *b* and *c*, Soil 1; *d* and *e*, Soil 2; *f* and *g*, Soil 3. For each soil, the first curve is for biotite from the upper horizon, the second for biotite from the lower horizon.

The *infra-red spectra* of weathered biotites from Soils 2 and 3 and of the fresh biotite are shown in fig. 2. The biotite from Soil 2 contains a well-crystallized kaolinite with OH-stretching absorption bands at 3697, 3669, 3652, and 3620 cm^{-1} (Russell and Farmer, 1964) and also gibbsite with OH bands at 3536 and 3479 cm^{-1}. A more poorly crystallized kaolinite is present in the biotite from Soil 3, as indicated by the absence of the 3669 and 3652 cm^{-1} bands, but gibbsite was not detected. The latter sample also has a rather broad OH-stretching band near 3575 cm^{-1}, probably due to chloritic interlayers shown to be present in some flakes by X-ray evidence. Both weathered biotites show a rather broad low-frequency OH-stretching band of the type that has been associated with allophanes (Mitchell *et al.*, 1964).

The *cation-exchange capacities* of the weathered biotite flakes from the

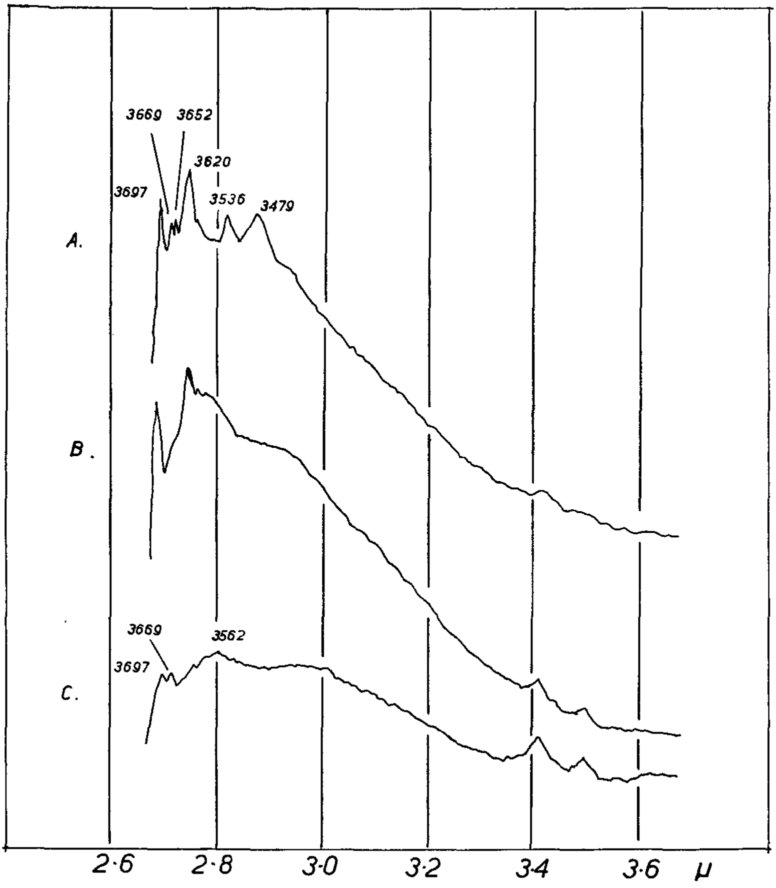

FIG. 2. Infra-red absorption curves for: A, weathered biotite from Soil 2; B, weathered biotite from Soil 3; C, fresh biotite.

upper and lower parts of all three soil profiles vary between 23·2 and 69·6 m. eq/100 g.

Soil No. 1		*Soil No. 2*		*Soil No. 3*	
12–15″	34·2 m. eq.	13–15″	24·9 m. eq.	3–6″	23·2 m. eq.
20–24″	28·8	20–23″	50·1	20–24″	69·6

Since the cation-exchange capacity of unweathered biotite is only 9 m.eq/100 g it appears that weathering has induced a considerable increase. Probably in view of their abundance the weathered biotites account for a large proportion of the total soil exchange capacity. It may be noted that the cation-exchange capacity of the weathered flakes from Soils 2 and 3 shows a marked increase in the lower horizons, but more work is needed before it can be established whether this is a feature of general significance.

Optical data. The weathered biotites were studied optically in the light fine sand fractions and also in slides cut from impregnated soils. Study by the former method is confined to the preferentially oriented (001) cleavage surfaces. However, sections cut from impregnated soil blocks do not suffer this disadvantage and here the biotite may be studied in a variety of attitudes.

The (001) cleavage sections are usually pale yellowish brown, non-pleochroic and of low birefringence. The β refractive indices of the weathered flakes are considerably lower than the indices of the fresh unweathered biotite. The fresh biotite had β 1·625, while the weathered samples had: Soil 1, 1·583 to 1·588; Soil 2, 1·579 to 1·583; Soil 3, 1·579 to 1·590. This accords with the general findings of many previous workers (e.g. Denison *et al.*, 1929; Walker, 1949). In convergent light the cleavage flakes show a biaxial negative interference figure with a small optic axial angle ranging from 5° to 15° approximately. The figure may often be distorted and its definition poor. The surfaces of the biotites frequently show micromorphological features comparable to those described by Raman and Jackson (1965) in their electron-optical study of mica surface morphology during laboratory weathering. These include various discontinuities such as curling and scrolling of mica layers and edges and fractures oriented at 120° angles (plate XIII, *a–d*). From plate XIII, *e*, which shows the characteristic appearance of many of the mica flakes between crossed nicols, it is clear that different parts of the same crystal are in different optical orientations. This probably results from the folded and discontinuous nature of the mica layers and perhaps also from the more acute internal disorder arising from the complex mosaic weathering of mica as envisaged by Jackson (1965).

The edges of the weathered biotites are seen in thin sections of impregnated soil blocks. The most prominent feature is the way the flakes are split and opened out along the cleavage planes (pl. XIII, *f* and *g*). Occasionally, these spaces are occupied by a mineral showing low relief and birefringence and a length-slow orientation. Almost certainly these are the phases of kaolinite or gibbsite or both detected by X-ray diffraction, d.t.a., and infra-red analysis, which may now be referred to as zones. Because of their low birefringence these zones may be more easily discerned with the gypsum plate, which, with the coincidence of the slow vibration directions, causes them to stand out as vivid blue streaks in sharp contrast to the biotite. Invariably the slow directions of the zones and of the biotite coincide indicating the controlling influence of the host mineral.

Discussion

From the results quoted it is clear that the weathering of biotite involves expansion in the *c* direction, interlayer alumination, formation of gibbsite zones, and formation of kaolinite zones. The natural weathering of biotite to vermiculite involving a lattice expansion from 10 Å to 14 Å was shown by Walker (1949). This transformation proceeds essentially through the replacement of interlayer potassium by other ions and by water, together with such changes as oxidation of octahedral iron. Jackson (1963*a* and 1963*b*) has described the way in which interlayer spaces of expanded 2:1 layer silicates in soil clays may be aluminated and the interlayer alumination of the weathered biotites described above may have proceeded in a comparable manner. Removal of potassium with concurrent lattice expansion provides negative surfaces on to which positively charged hydroxo-aquo-aluminium ions become attached so binding the sheets together. Eventual polymerization of this complex then creates an intergrade vermiculite-chlorite. In this particular instance the aluminium is probably derived from hydrolysis of the labradorite of the parent material—which would also result in the release of some silica—and indeed microscopic examination of this mineral in the fine sand fraction clearly shows features characteristic of solution. Edges invariably present an etched or ragged appearance, usually due to differential solution of the feldspar along adjacent twin lamellae.

The formation of gibbsite zones within the biotite crystals may be favoured by the tendency of the biotite to split and open out during the course of weathering (Pl. XIII, *f* and *g*). It is significant in this respect that Slaughter and Milne (1960) were able to synthesize gibbsite, attached to one surface only, in the interlayer spaces of swollen montmorillonite. Barnhisel and Rich (1963) also achieved the formation of gibbsite in the laboratory under acid conditions from aluminium-interlayered montmorillonite. Thus it seems likely that the splitting of the biotite flakes during weathering presents fresh surfaces for the organization and precipitation of introduced hydroxy-aluminium together with the space in which discrete zones of gibbsite can grow.

Kaolinite zones could grow in the biotite crystal in a like manner. On the other hand, a topotactic weathering transformation of expanded 2:1 layer silicate clays has been suggested by several workers. For example, Glenn *et al.* (1960) considered that montmorillonite weathered to kaolinite via the intermediate stage of intergradient vermiculite-chlorite. Such a transformation involves complete interlayer alumination of the montmorillonite and also inversion of silica tetrahedra. The evolution of kaolinite is thus envisaged as proceeding along a 2:1–2:2–1:1 pathway. A similar topotactic change from biotite to kaolinite, however, would be drastic since it would involve: formation of a complete gibbsite sheet in the interlayer spaces; conversion of the octahedral layer from tri- to dioctahedral with the expulsion of iron and magnesium and the introduction of aluminium; introduction of Si^{4+} ions into the tetrahedral layer; and tetrahedral inversion. Furthermore, these changes would occur only at particular parts of the biotite structure.

In general the building up of kaolinite zones by epitaxial crystallization affords a more acceptable explanation. Thus initial weathering opens out the biotite flakes along the cleavage planes. The exposed surfaces then act as templates that control the orientation of the kaolinite during subsequent growth presumably from an alumina–silica gel. The precise chemical and physical conditions that could induce such a crystallization are unknown and recent work by De Kimpe *et al.* (1964) indicates kaolinite synthesis at low temperatures and pressures to be exceptionally difficult and that it is essential for six-fold co-ordinated aluminium to be stabilized; in the presence of silica this can only be achieved at low pH. The same authors also found that seeding an alumina-silica gel with crystalline muscovite brought about an appreciable yield of kaolinite, although this occurs only at slightly elevated temperatures and pressures.

In a general analysis of the pedogenic weathering of micas, including biotite, Jackson (1963 *a*, *b*) proposed the following sequence of reactions, most of which occur in the clay-size fraction of the soil:

Pedogenic
Mica → Vermiculite → 2:1–2:2 14 Å ⟶ Al ⟶ Kaolinite
(Biotite) intergrade Chlorite and Halloysite

The above sequence implies that change to each succeeding phase is complete. However, it has been shown previously that single crystals of the weathered biotite up to 1 mm in diameter may contain several different phases and that therefore the reaction outlined above is inapplicable in this instance. Although the exact relationship between aluminium interlayering and kaolinite and gibbsite zonation in biotites is unknown, it seems probable that their formation was relatively rapid and contemporaneous. Thus biotite weathered in this manner has been found in only slightly disintegrated rock where visually the flakes look fairly fresh. Provided chemical conditions are suitable, therefore, there seems to be no reason why clay minerals such as kaolinite or gibbsite, which are usually indicators of relatively intense weathering, should not crystallize epitaxially almost at the inception of the decomposition of a mineral.

The breakdown of the Strathdon biotite may be envisaged as occurring as follows: the first stage involves expansion of the biotite to vermiculite with some concurrent interlayer alumination; subsequently, there is a build up of interlayer aluminium thereby creating an aluminous intergrade vermiculite-chlorite, together with splitting along the cleavage planes permitting epitaxial crystallization of kaolinite and gibbsite. Further weathering could result in the destruction of the intergradient mineral leaving pseudomorphs of kaolinite and gibbsite; it is noteworthy that kaolinite pseudomorphs after biotite have been found by the writer in the coarse sand fraction of Soil 3. These reactions may thus be formalized:

Biotite (4) → Vermiculite (8)
Intergrade vermiculite-chlorite (9) →

Intergrade vermiculite-chlorite (9) → Kaolinite (10)
Kaolinite (10) Gibbsite (11)
Gibbsite (11)

where the bracketed numbers represent weathering indexes as proposed by Jackson *et al.* (1948). Kaolinite and gibbsite are the final products of weathering because relatively they are the most resistant.

Conclusions

The present study has shown by a variety of techniques that the biotites in some freely drained soils near Strathdon, Aberdeenshire, weather to a complex polycomponent assemblage of aluminous vermiculite-chlorite with zones of kaolinite and gibbsite. Since flakes of this nature up to 1 mm in diameter have been found in only slightly disintegrated rock it is considered that these changes occurred relatively quickly. The kaolinite and gibbsite zones crystallized epitaxially within cleavage cracks and do not represent intensely altered parts of the biotite structure. Walker (1949) showed that under intermediate drainage conditions biotite changed to vermiculite whilst MacEwan (1953) discovered a biotite-to-montmorillonite transformation in some poorly drained soils. It is possible that the changes described above are characteristic of a freely drained environment, although the precise conditions which favour such weathering are at present unknown.

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EXPLANATION OF PLATE XIII

FIG. *a*. Weathered biotite from Soil 1 showing curled edges and typical surface morphology. ×650

FIG. *b*. Weathered biotite—Soil 1—showing surface scrolling of layers. ×250.

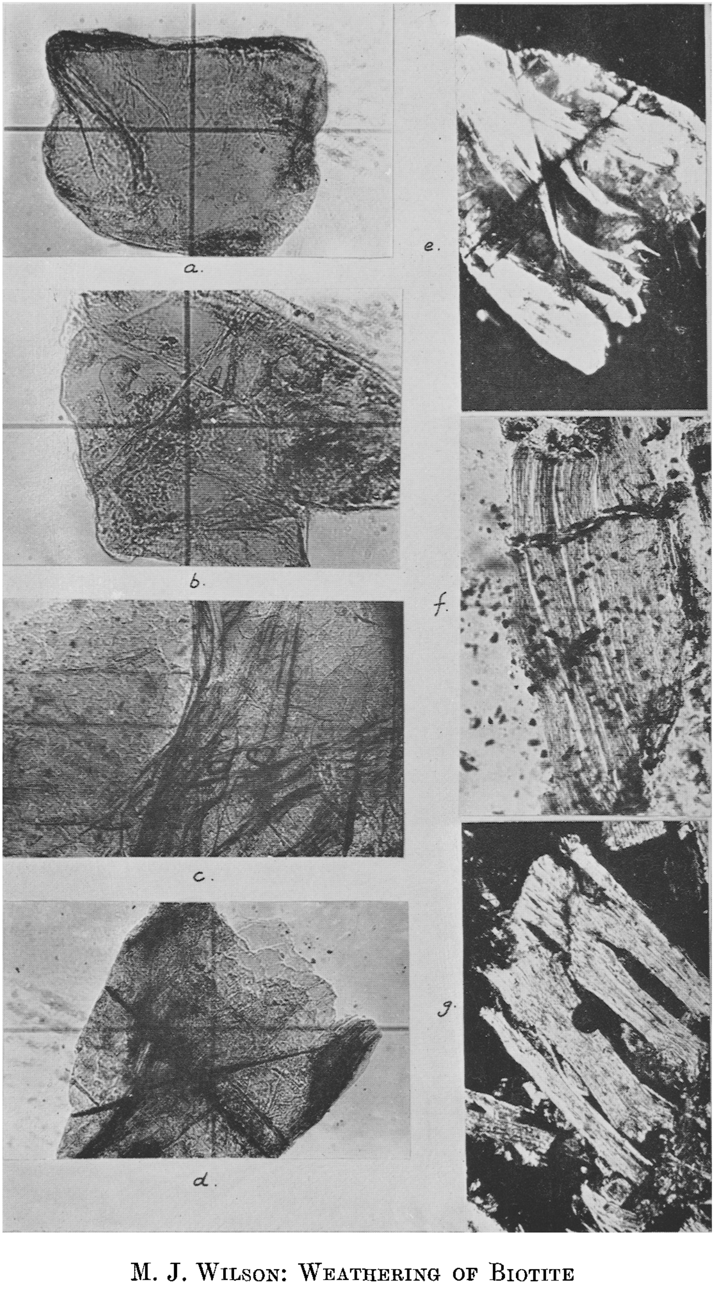
FIG. *c*. Weathered biotite—Soil 2—showing surface irregularities and cracks. ×250.

FIG. *d*. Weathered biotite—Soil 2—showing scrolls, surface irregularities and cracks at 120° angles. ×250.

FIG. *e*. As above—Characteristic appearance of weathered biotites between crossed nicols.

FIG. *f*. Weathered biotite—Soil 2—split and opened out along cleavage planes. ×250.

FIG. *g*. Weathered biotite—Soil 2—showing exfoliated appearance. ×250, crossed nicols.



M. J. WILSON: WEATHERING OF BIOTITE