VISIBLE CHANGES IN MACRO MICA PARTICLES THAT OCCUR WITH POTASSIUM DEPLETION*

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ABSTRACT

Mica particles approximately 10 or 25 mm square and 0.5 mm thick were placed in NaCl–NaTPB solutions to make visual observations of the changes that occur in micas when the interlayer K is replaced by Na. Samples of muscovite, biotite, phlogopite, lepidolite, and lepidomelane were used, and the effects of different degradation periods were photographed.

An increase in the thickness of the particles due to basal planes splitting apart was observed with all micas. This exfoliation released interlayer K and in some cases caused the particles to cleave into separate flakes. Lepidomelane particles remained intact despite a 20-fold increase in thickness in 7 days. Even muscovite and lepidolite exfoliated and cleaved, but much longer degradation periods were needed.

There was a distinct change in the color of the dark biotite, phlogopite and lepidomelane particles when K was removed. Therefore, the initial stages of K depletion at holes, scratches, and edges of the particles were easily followed. As the degradation of the mica particles progressed, however, the color of the mica became a less reliable index of the stage of K depletion. Visual evidence of K depletion at the edges of particles was also obtained with muscovite, but not with lepidolite.

Transverse sections of 25-mm particles of K-depleted biotite were photographed to show the edge expansion that occurred when interlayer K was replaced by Na.

INTRODUCTION

Concepts regarding the replacement of interlayer K in micaceous minerals have been developed with ground and naturally fine-grained samples of mica (Scott and Smith, 1966). As a result, small particles of highly degraded mica have been available and used to determine the mineral alterations that occur with a loss of K. For optical observations of the changes in K-depleted mica, however, larger particles have been degraded and examined with a microscope (Mehmel, 1938; Mortland, 1958; Rausell-Colom *et al.*, 1965; Weiss, Mehler, and Hoffman, 1956). The use of even larger particles for a direct observation of these changes has been largely precluded by limitations in the K extraction methods heretofore available. Now it is known that the

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interlayer K, even in muscovite, can be replaced effectively with NaTPB solutions (Reed and Scott, 1966). Therefore, large mica particles were placed in NaTPB solutions to determine, by direct observation, the changes that occur in micas when the interlayer K is removed.

MATERIALS AND METHODS

Large sheets of muscovite, biotite, lepidomelane, and two phlogopites (referred to here as I and II) from Ontario and of lepidolite from Colorado were obtained from Ward's Natural Science Establishment. These sheets were split until they were only 1 to 2 mm thick, cut with a razor blade to obtain square particles of about 10×10 mm or 25×25 mm, and finally split again to reduce the particle thickness to about 0.5 mm. Most of the thin, square particles were prepared with dry mica in air, but a few were cut and split under water for specific comparisons. For simplicity, the prepared mica particles will be referred to as 10-mm and 25-mm particles. Several particles of both sizes were selected at random for the experiments to ensure the inclusion of effects from variations in the particles in regard to edges, surface cracks, holes, etc.

Individual 10- and 25-mm mica particles were placed in 10 ml 1 N NaCl–0.2 N NaTPB–0.01 M EDTA solution and stored at 25°C. More solution was added as needed to keep the particle completely immersed. After specific contact periods, the degraded particles or portions of these particles were removed, prepared in the manner described with each experiment, and photographed.

The amounts of K extracted from single particles in the NaTPB solution for specific periods and the amounts of K in various degraded mica particles were determined by the procedures that have been used with finely divided mica samples (Reed and Scott, 1966; Scott and Reed, 1965). For the extracted K determinations, the KTPB was destroyed with a boiling NH_4Cl–$HgCl_2$ solution, whereas the degraded particles for total K analysis were washed free of KTPB with a 1:1 acetone-water solution that contained NH_4Cl and then digested in HF–$HClO_4$. The K brought into solution by these treatments was determined by flame emission.

RESULTS AND DISCUSSION

Exfoliation

A rapid increase in particle thickness was observed when the biotite, phlogopite, and lepidomelane particles were placed in the NaTPB solution. This increase was particularly evident during the first day of treatment, but further increases were observed for 3 to 7 days. Much longer contact periods did not thicken the muscovite and lepidolite particles appreciably. Therefore, the lateral edges of the various micas were photographed after 7 days' treatment to show this initial effect of K depletion.

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Plate 1A shows the increase in particle thickness observed with a 10-mm biotite particle, and Plate 1B, consisting of two photographs taken with the same degree of magnification, shows the relative change observed with 25-mm particles of different micas. To obtain these photographs, the treated particles were separated from the NaTPB solution, rinsed with water to remove the salts that tend to obscure the surface, and kept wet while supported in air. With this treatment, the expanded 10-mm biotite particle split into several flakes which were stacked for the photograph. There was also a slight decrease in the thickness of the expanded particles when they were taken out of the water. Thus, Plates 1A and 1B do not provide a quantitative measure of the maximum expansion observed. By photographing the expanded 25-mm lepidomelane particle under water, however, the maximum expansion attained with this mica is shown in Plate 1C.

An estimate of the amount of expansion that occurred in the different micas was obtained by measuring the wet expanded particles in air. An 8-fold increase in thickness was observed with both the 10-mm and the 25-mm biotite particles. A similar increase was observed with phlogopite II. On the other hand, there was a 16-fold increase in the thickness of the lepidomelane particles and only a very small expansion at the edges of the muscovite and lepidolite particles.

The expanded lepidomelane particle under water (Plate 1C) was 17 mm thick. Since the original particle was approximately 0.8 mm thick, this means that there was a 20-fold expansion without the particle being split into separate flakes. Several wide separations of adjacent layers and interlayer islands of KTPB can be seen in this particle, but the cleavage planes apparently did not extend across the particle. A comparison of the photographs in Plates 1B and 1C of the same lepidomelane particle shows that the wide interlayer separations disappeared when the particle was taken out of water. This accounts for the reduction in particle thickness that was observed in the preparation of the photographs in Plates 1A and 1B.

Mica particles that were prepared by cutting and splitting mica sheets under water were also placed in NaTPB solutions for a week. These particles expanded like those shown in Plate 1. On the other hand, mica particles that were prepared in water or in air did not expand when they were placed in water, NaCl, or NaCl–EDTA solutions for a week. Thus, it has been concluded that the initial expansion observed with mica particles in NaTPB solutions was due to a replacement of interlayer K, not to the method of particle preparation. Apparently, several silicate layers in the particle were split apart by the expansion force from the initial exchange of Na for K at the edges of the particle. This means the mica particle exfoliated. As a result, there should have been a rapid release of K by this exposure of interlayer surfaces.

The amounts of K that were released by 10-mm mica particles in 10 ml of 1N NaCl–0.2N NaTPB–0.01M EDTA for 1, 3, and 7 days are reported in Table 1. The total and NaCl extractable K values for these particles are also

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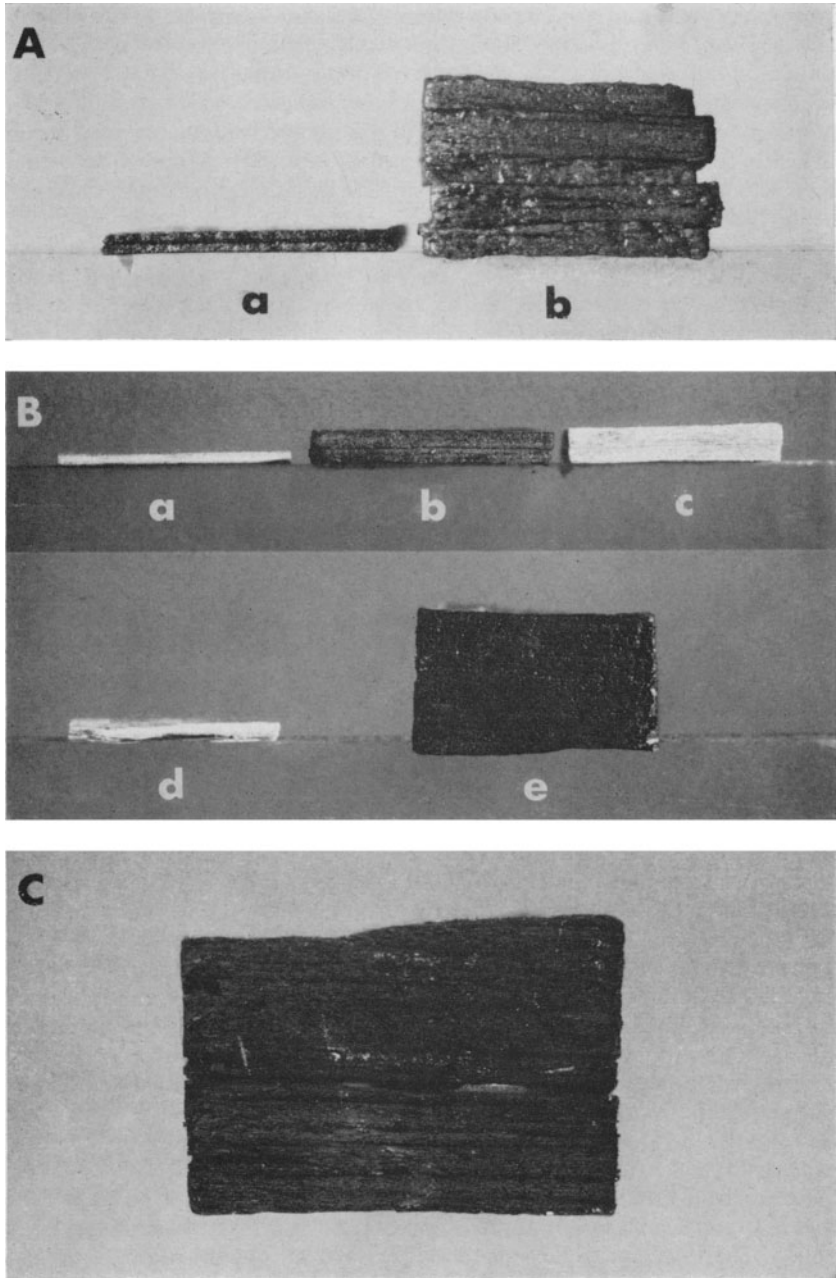


PLATE 1. Edges of wet mica particles after 7 days in NaTPB solutions. A. 10-mm biotite particles in air: (a) original and (b) treated. B. 25-mm particles in air: (a) muscovite, (b) biotite, (c) phlogopite II, (d) lepidolite, and (e) lepidomelane. C. 25-mm lepidomelane particle in water.

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TABLE 1.—K EXTRACTED FROM MACRO MICA PARTICLES IN NaCl–NaTPB SOLUTIONS FOR SHORT PERIODS

10-mm mica particles	Total K meq/100 g	K extracted (meq/100 g)			
		NaCl	NaCl–NaTPB		
		7 days	1 day	3 days	7 days
Muscovite	222	0.3	2.0	4.1	4.9
Biotite	199	1.3	8.4	12.1	15.2
Phlogopite II	220	0.6	8.2	10.4	15.8
Lepidolite	214	0.2	1.8	2.3	2.4
Lepidomelane	198	2.2	14.6	36.1	39.6

given. The extracted K data were obtained with single particles, and duplicate experiments, and are presented only to show that there was a rapid release of K when the mica particles exfoliated. Nearly 20% of the K in lepidomelane was released in this manner. Therefore, this release of K by exfoliation must be considered along with edge weathering in the kinetics of interlayer K exchange. The small amount of K extracted from the muscovite and lepidolite particles is consistent with the observation that these micas did not exfoliate during the first week.

Edge Weathering

A gradual increase in the amount of KTPB precipitate on or around the mica particles was observed when the particles were kept in the NaTPB solution for progressively longer periods. This meant a release of interlayer K continued to occur after the initial period of exfoliation and K release by layers. To determine the site of this K release, particles or cleavage flakes of particles were separated from the NaTPB solution and air dried. In this manner, the zones of K release and precipitation were accentuated by the presence of the dry salts. The results obtained with biotite particles are shown in Plate 2.

These photographs show that there was a concentration of salt at the lateral edges of the particles, at holes and cracks in the basal surfaces, and at the edges of incomplete silicate layers. Since the K replaced by Na would be precipitated as soon as it reached the exterior of the particle, these sites of KTPB accumulation show the release of K occurred only at points where the interlayer region was exposed to the extracting solution. These results, therefore, constitute visual evidence for the model of edge weathering in micas that was suggested by Bray (1937) and later substantiated by optical evidence (Mortland, 1958; Rausell-Colom *et al.*, 1965) and a kinetic study of K release (Reed and Scott, 1962). The photographs in Plate 2, however, have verified this model by a direct observation of the precipitated K.

The photographs of a corner of a dry, 3-month degraded, 10-mm biotite

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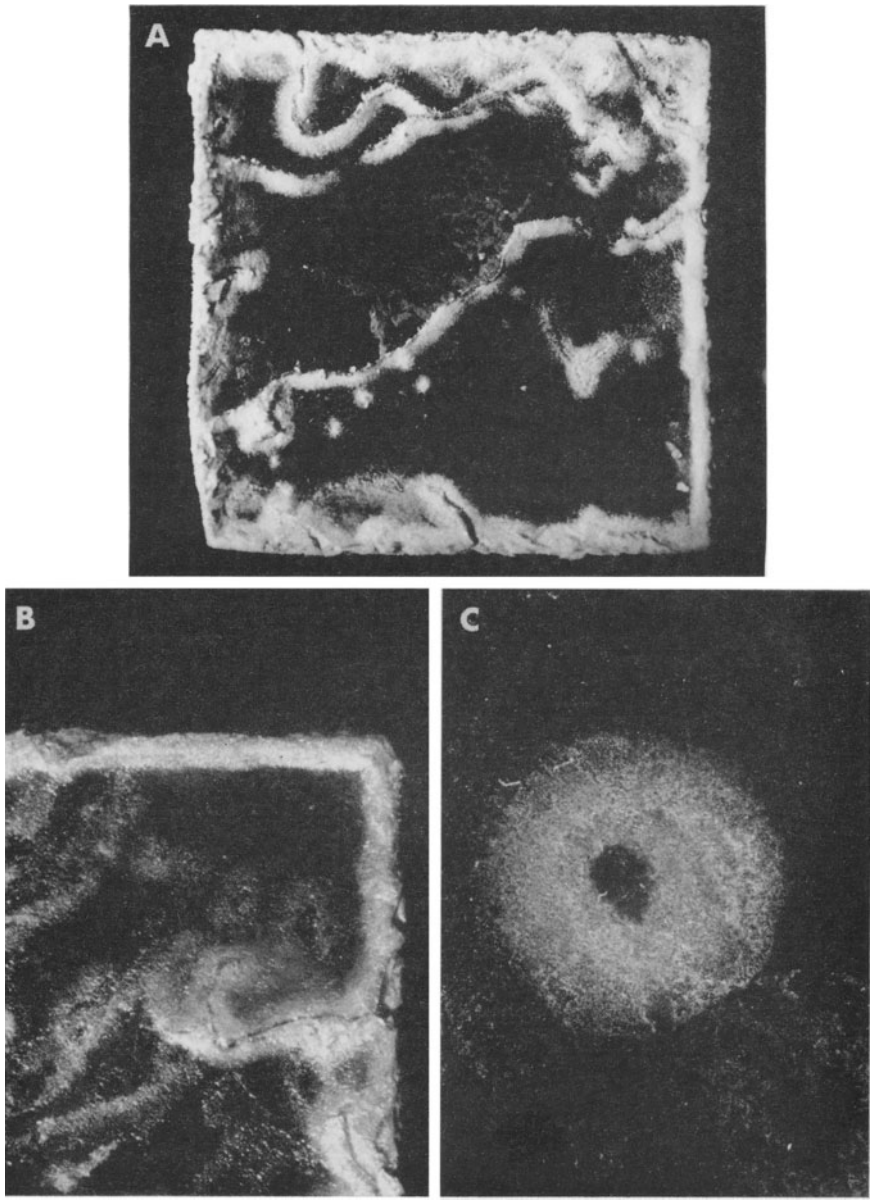


PLATE 2. Exposed basal surfaces of air-dry flakes of NaTPB-treated biotite. A. 10-mm flake treated 3 months. B. Corner of 10-mm flake treated 3 months, magnified 12×. C. Pin hole in 25-mm flake treated 18 months, magnified 12×.

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particle (Plate 2B) and of a pin hole in the basal surface of a dry, 18-month degraded, 25-mm biotite particle (Plate 2C) provide a detailed view of the zones of K depletion. They also show the shortcomings of this method of following the progress of K depletion. The zone of K depletion at the corner is sufficiently distinct to show the sharp corner maintained by the undegraded portion of the particle and the location of the weathering fronts that are parallel to the edges of the particle. On the other hand, the dry salts on the surface of the particle tend to obscure the zone of K depletion around the cracks. This difficulty with salts increased with the stage of degradation and the amount of KTPB.

Similarly, the pin hole in the mica surface (the dark spot in the center of the white area of Plate 2C) was covered with salt and, therefore, not visible at first. To obtain this photograph, the salt was removed with a damp cloth. This treatment enlarged the hole slightly, but it showed the hole was extremely shallow. However, this hole provided an access for the exchange of Na for the K in a thin outer layer of the particle, and a circular zone of K depletion developed. The dark color of the hole is due to the mica layers below. The light color of the surrounding circular zone was presumed to be salt at first, but was found to be the color of the thin outer layer of degraded mica.

Since mica alterations were often difficult to see in the presence of dry salts, some degraded mica particles were washed in water to remove the salt and photographed while immersed in water. The photographs in Plate 3 of 10-mm biotite and lepidomelane at different stages of K depletion were obtained in this manner.

A distinct change in the color of these dark mica particles was observed when the particles were placed in NaTPB solutions. This color change started at the edges of all lateral breaks that exposed interlayer regions to the extracting solution and then progressed across the particle. The light-colored portions of the partially degraded particles were expanded and K depleted. The boundary between the light and dark colors, therefore, marked the extent to which the interlayer K was replaced by Na. This boundary provided a means of following the loss of interlayer K, just as other boundaries have indicated interlayer changes (Rausell-Colom *et al.*, 1965; Walker, 1956; 1959; Weiss, Mehler, and Hoffman, 1956).

In the early stages of K depletion, the boundary was generally quite distinct and parallel to cleanly cut lateral edges. This was the case at the top edge of the biotite particle in Plate 3A, which also shows the irregular and less well-defined weathering front that occurred when the edges of the particle were ragged. An advanced stage of degradation was achieved in 7 weeks with the 10-mm lepidomelane particle (Plate 3B). In this particle, the boundary was not sharp, but it showed the rate of K replacement was uniform in all directions. On the other hand, the 6-month degraded biotite particle in Plate 3C shows the situation frequently encountered with mica particles that have cracks and a slower rate of K release. In this case, the irregular

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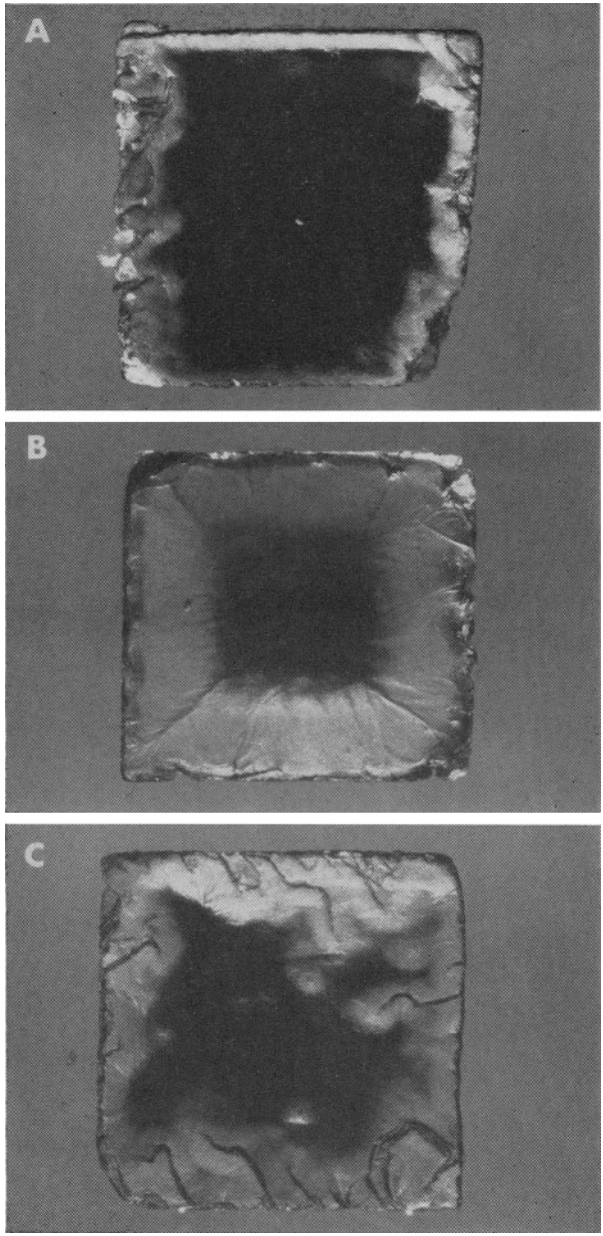


PLATE 3. Edge weathered, 10-mm mica flakes in water. A. Biotite treated 15 weeks. B. Lepidomelane treated 7 weeks. C. Biotite treated 6 months.

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front and the poorly defined color boundary made it essentially impossible to estimate the stage of K depletion.

Particle Disruption

Several physical changes in the mica particles occurred when the NaTPB treatment was continued for long periods. Some of these changes are shown in Plate 4. For these photographs, the particles were sprayed with water to remove the salts and either immersed in water (A, B, and C) or kept wet while supported in air (D, E, and F).

The muscovite and lepidolite particles tended to remain intact and to resist change. Plate 4A shows a 10-mm muscovite particle that was degraded for 3 months. In this period, there was no evidence of exfoliation or cleavage, only a slight expansion of the edges of the particle. In 6 to 9 months, however, a separation of layers and an increase in particle thickness was observed, even with muscovite and lepidolite.

The initial expansion of biotite, phlogopite, and lepidomelane particles gave rise to different amounts of cleavage in the first week. The 10-mm biotite particle in Plate 1A, for instance, had split into several flakes which were stacked in air for the photograph. In water, however, the flakes tended to separate as shown by the 3-month degraded, 10-mm phlogopite I particle in Plate 4C. This separation of flakes was observed particularly with the 10-mm particles. Often, the 25-mm particles would remain intact even though the particle expanded extensively and parts of adjacent layers were quite far apart (Plate 1C). Cleavage flakes produced during the initial period of exfoliation did not cleave further as the K-depletion process continued.

The cleavage of partially degraded mica particles was most evident with biotite, phlogopite, and lepidomelane. However, some cleavage was observed with muscovite and lepidolite particles that were degraded for 6 to 18 months. A 25-mm muscovite particle separated into three flakes in an 18-month degradation period. Several 10-mm particles have split into two flakes in shorter periods.

Plate 4B shows a 10-mm biotite particle that was degraded for 3 months. In addition to splitting into separate flakes, this particle crumbled into several irregular pieces. This type of disintegration of particles was observed only with biotite and lepidomelane. Since these micas did not crumble as much when uniform, crack-free particles were used, this disintegration was probably due to imperfections in the specimens. Nevertheless, the disruptive forces breaking down the particles must have been great to cause the lateral breaks that can be seen in the silicate layers of the phlogopite particle in Plate 4C.

Experiments with $< 50\ \mu$ samples of all the micas have shown the basal spacing of the micas increases from 10 Å to 12.3 or 15 Å when the interlayer K is replaced by Na (Scott and Smith, 1966; unpublished data for lepidolite and lepidomelane). Consequently, the exchange of Na for K at the lateral

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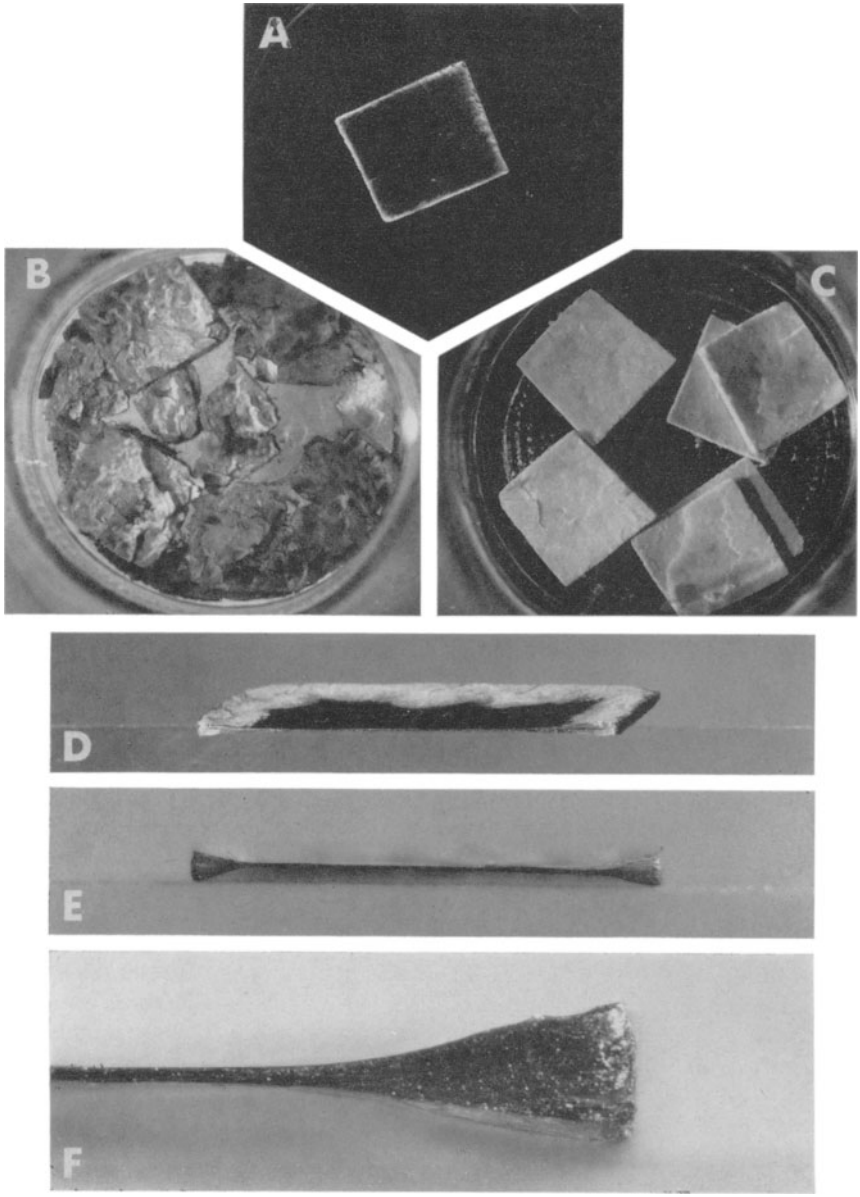


PLATE 4. Disruptive effects of NaTPB treatment. Degraded 3 months, 10-mm particles in water: A. Intact muscovite. B. Fragmented biotite. C. Cleaved phlogopite I. Degraded 18 months, 25-mm biotite flakes in air: D. Partially dry segment. E. Wet transverse section. F. Wet end of transverse section.

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edges of the macro mica particles should have given rise to an expansion of the edge. This edge expansion is shown in the photographs of a 25-mm biotite flake in Plates 4D, 4E, and 4F. To obtain these photographs, one of the cleavage flakes of a biotite particle that had been degraded for 18 months was rinsed in water to remove the salts and cut with a razor blade while wet to obtain a transverse section. The cut sections were supported in air and partially dried for photograph 4D or kept wet for photographs 4E and 4F.

The oblique view of the cut flake in Plate 4D shows that there was a close relationship between the expanded edge and the zone of K depletion indicated by the light surface color. This particle was partially dried to increase the contrast between the light and dark colors of the basal surface. However, this drying lightened the color of the face of the transverse cut and reduced the sharpness of the bends in the silicate layers. For these features, reference should be made to the photographs of wet flakes in Plates 4E and 4F. Unlike the basal surface of the particle, the transverse section had a uniform, dark color. The silicate layers were bent quite sharply by the expansion of the edges.

By measuring the thickness of the center and the edge portions of the transverse section in Plate 4F, it was calculated that there had been an 8-fold expansion at the edge. This was much more than could be accounted for by a 5 Å increase (10 to 15 Å) in basal spacing. Therefore, many of the layers must have been widely separated in these weathered edges just as larger portions of layers were separated in the initial exfoliation of the particles. The center of this flake, which was only one of several cleavage flakes arising from the original biotite particle, was 0.15 mm thick. Apparently, the flakes needed to be quite thin to withstand so much edge expansion without further cleavage.

Comparison of Micas

Photographs of 10-mm particles and cleavage plates of micas that were degraded for 3 months are shown in Plate 5. These mica samples were rinsed with water to remove the salts and photographed while immersed in water to show the relative amounts of edge weathering attained with each.

The muscovite (Plate 5A) and lepidolite (Plate 5D) particles did not cleave during this degradation period; therefore, the entire particle was photographed. Being translucent, the muscovite particle assumed the color of the background. This proved useful because the light, weathered edge of this particle was difficult to see until the rest of the particle was darkened with a black background. There was no evidence of edge weathering in the lepidolite particle. The irregular light and dark coloring of the particle in Plate 5D existed in the original particle. This coloring would make it difficult to see edge weathering, but even 6 months' degradation did not produce a visible effect on the edges. Instead, the general color of the surface became lighter.

The biotite, phlogopite, and lepidomelane photographs show the color

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changes and edge weathering observed with flakes that cleaved from the original particle. With these micas, there was an obvious change in color as edge weathering progressed. In 3 months, the biotite (Plate 5B) and phlogopite II (Plate 5C) particles exhibited similar amounts of edge change and more than muscovite, as expected. On the other hand, the particle of phlogopite I (Plate 5F), which released K at nearly the same rate as biotite in experiments with $<50\ \mu$ samples (Scott and Smith, 1966), underwent a complete change in color. The particle in Plate 5F was originally as dark as the inner core of the biotite particle in Plate 5B. The lepidomelane particles reached an advanced stage of edge weathering in 7 weeks as shown by Plate 3B. By 3 months, the color of the entire lepidomelane particle had changed, but it is evident from Plate 5E that the center portion of the particle had not yet expanded. Also, it will be shown later that both the phlogopite I and lepidomelane particles contained large amounts of K at this stage of degradation, despite the change in color.

Cleavage flakes from 25-mm particles of biotite and muscovite that were degraded for 18 months are shown in Plates 6 and 7. These flakes were rinsed in water to remove the salts and photographed while immersed in water. In 18 months the biotite flake (Plate 6) developed a wide band of color change and, presumably, K-depleted mica at the edges of the flake and at the edge of the incomplete silicate layer on the basal surface. This K-depleted portion of the biotite flake was expanded and soft enough to be dented (bottom and left side) when handled with tweezers. Since the muscovite cleavage flakes were very thin and translucent, it was difficult to photograph the changes in their edges. However, the flakes in Plates 6B and 7 show that there was a pronounced band of change wherever the silicate sheets terminated in a lateral break. A comparison of the muscovite and biotite flakes shows the striking difference in their rate of K release.

Residual K Estimates

Several degraded mica particles and flakes were analyzed to determine the relationship between the actual K content of the mica and the stage of K depletion estimated from the color boundary. The limited amount of alteration in muscovite and lepidolite particles eliminated them from this comparison. The loss of K from the 3-month degraded particles in Plates 5A and 5D was less than the experimental error of the K determination. The 18-month degraded particle of muscovite lost only 7 meq K/100 g. These small changes in total K are consistent with the small visible alterations in the particles.

The total K in the biotite and phlogopite II particles in Plates 5B and 5C was estimated from the dark central core in each and found to be 165 and 192 meq K/100 g, respectively. By analysis, there was 162 and 178 meq K/100 g in these particles. Thus, the visible and actual stages of K depletion were closely related. On the other hand, it was calculated that there was

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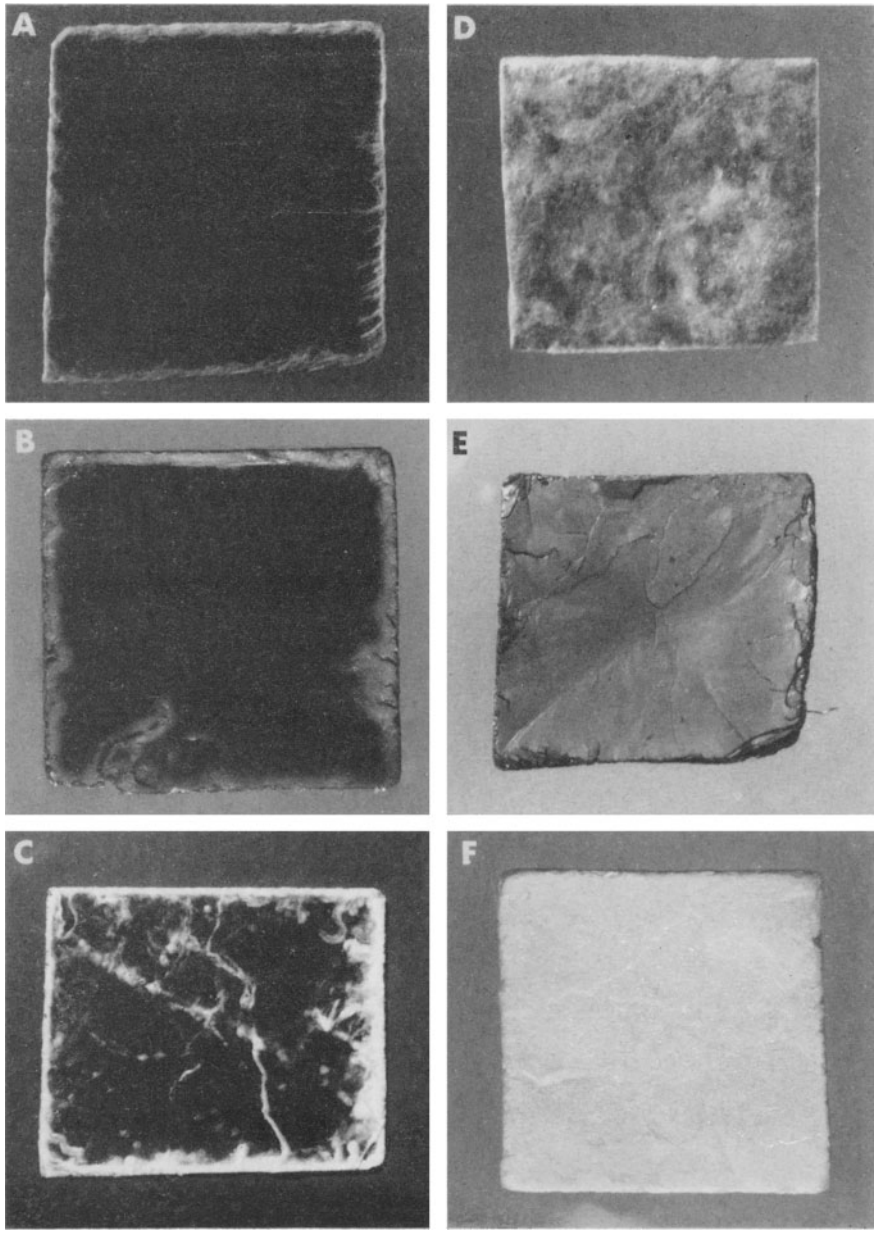


PLATE 5. Mica particles and flakes (10 mm) in water after 3 months in NaTPB solutions. A. Muscovite. B. Biotite. C. Phlogopite II. D. Lepidolite. E. Lepidomelane. F. Phlogopite I.

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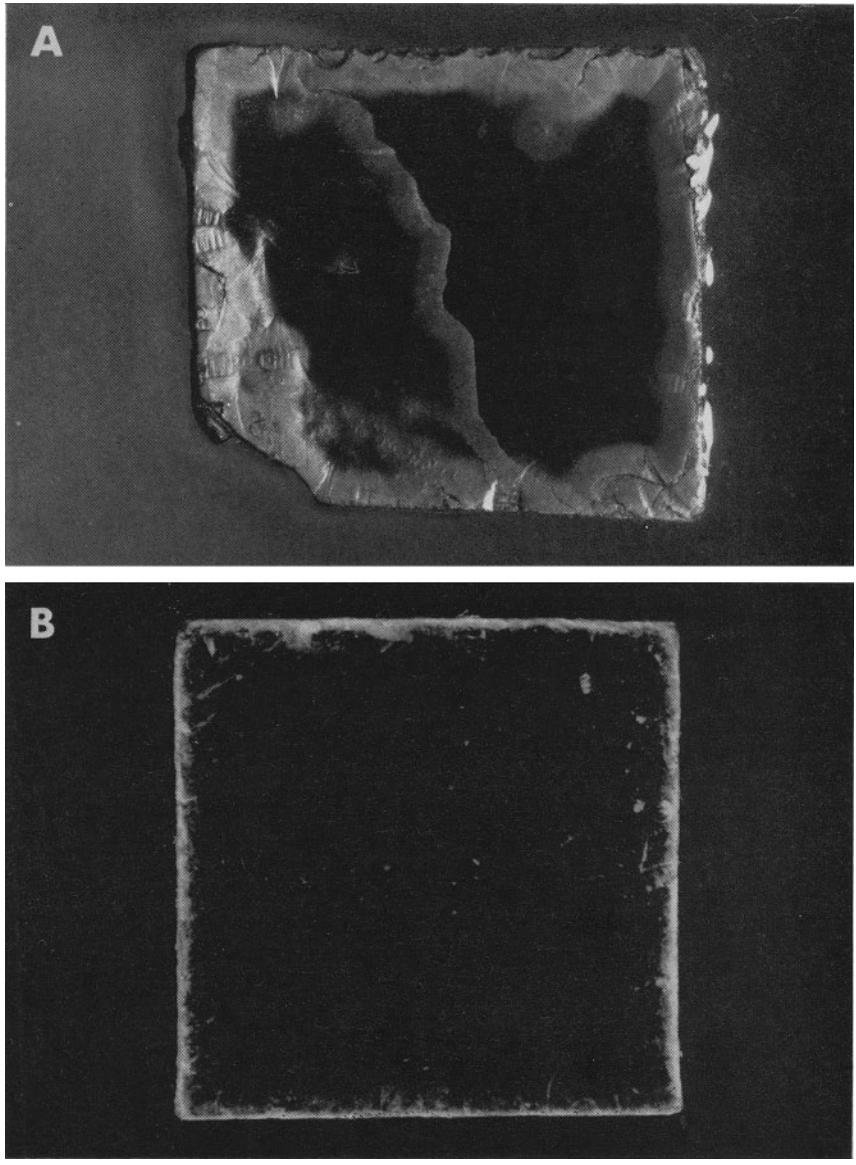


PLATE 6. Mica flakes (25 mm) in water after 18 months in NaTPB solutions. A. Biotite. B. Muscovite.

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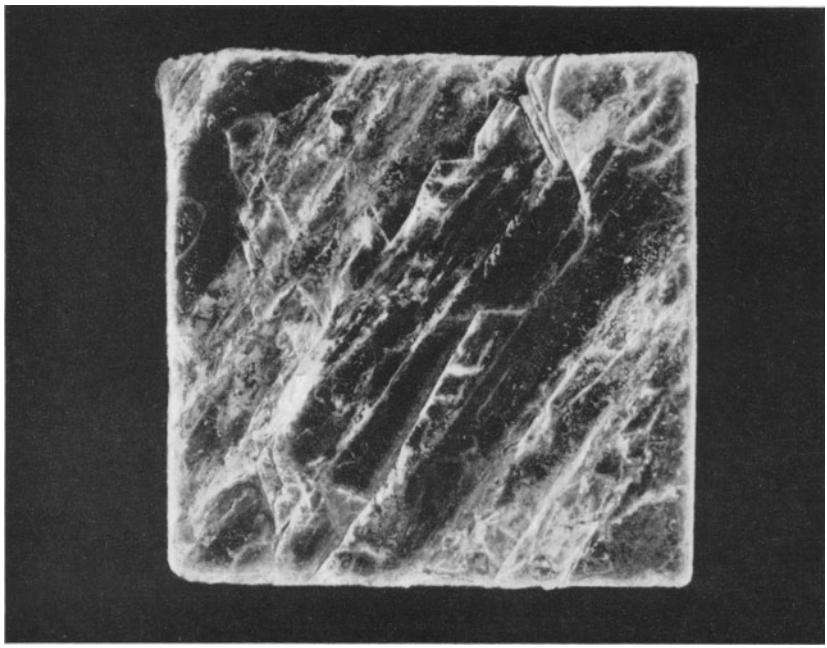


PLATE 7. Muscovite flake (25 mm) in water after 18 months in NaTPB solution.

only 48 meq K/100 g in the lepidomelane particle in Plate 3B when there was actually 107 meq K/100 g present. Also, with particles in advanced stages of K depletion, the central core of undegraded mica was often too irregular (Plate 3C) to calculate the remaining K or even absent (Plates 5E and 5F) when the particles still contained more than 65 meq K/100 g.

Discrepancies between the visible and actual stages of K depletion developed in different ways. Two of these situations are shown in Plate 8.

Three 10-mm particles of phlogopite I that were degraded for different periods are shown in Plate 8A. The original particle (*a*) contained 195 meq K/100 g and was very dark brown. After 1 month in the NaTPB solution, there was 154 meq K/100 g in the cleavage flakes (*b*) from this particle. The edges of this flake were expanded and light in color like other degraded mica particles, but the color of the rest of the particle was also much lighter. This change in the color of the whole particle continued without further evidence of an advancing weathering front from the edges. In 3 months all the original dark color had disappeared, but there was still 74 meq K/100 g in flake (*c*). The reason for this general loss of color by this mica is still not known. It did not occur in particles that were placed in water or in solutions that contained only NaCl or EDTA. A solution of NaTPB by itself did change the color of the particles. Thus, the color change appears to be related to a

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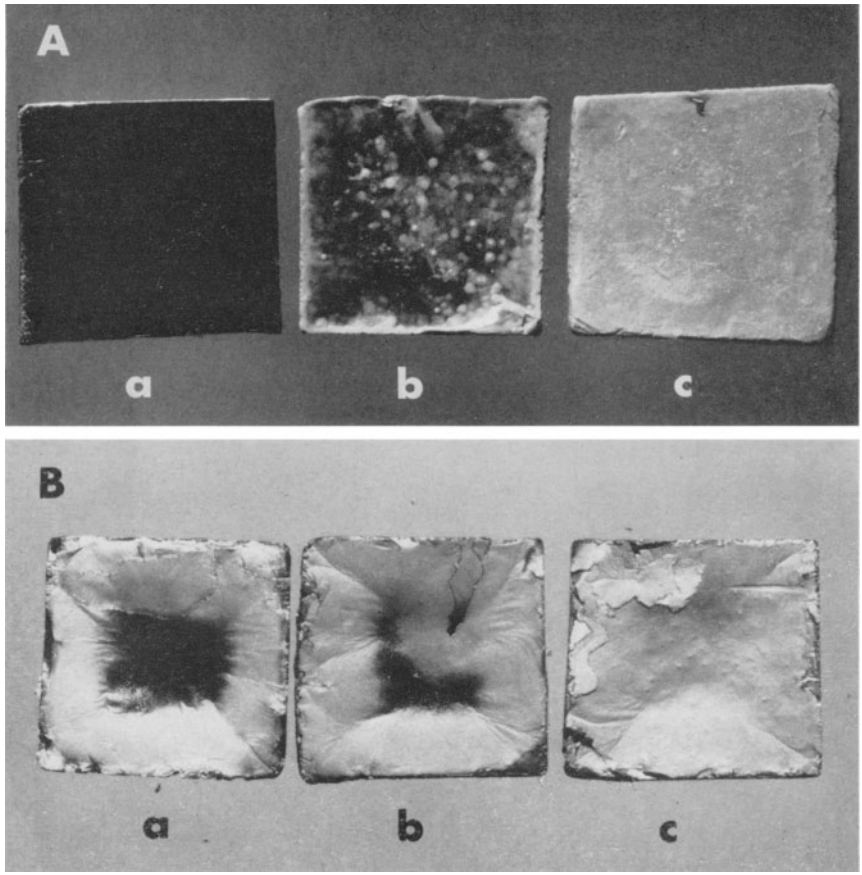


PLATE 8. Mica flakes (10 mm) in water showing deviations in color changes from NaTPB treatment. A. Phlogopite I: (a) original, (b) treated 6 weeks, and (c) treated 3 months. B. Lepidomelane flakes from same particle after 7 weeks' treatment.

loss of K, but some characteristic of the basal surface must be involved to obtain a simultaneous change across the surface.

Plate 8B shows three cleavage flakes from the same 10-mm lepidomelane particle that was degraded for 7 weeks. The dark central core in these flakes was obviously different, yet the K content of flakes *a*, *b*, and *c* was 82, 81, and 76 meq/100 g, respectively. The difference in appearance is due to a separation of the outer silicate layer of particles *b* and *c* from the layers below. When a portion of this outer layer was removed from the upper right corner of particle *b*, the dark color of the underlying layers of undegraded mica was

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visible. These observations illustrate the difficulty that can be encountered in following the progress of K depletion by color boundaries in the particle. They also suggest that the edge color change observed with K depletion was due to a separation of layers. This may also be the case with biotite.

It was frequently observed that the actual K content of a degraded mica particle was greater than the K content calculated from well defined zones of K depletion (e.g. the lepidomelane particle in Plate 3B). This was particularly the case with particles in advanced stages of K depletion. Therefore, it was concluded that there were appreciable amounts of K in the peripheral weathered zone. The expanded edges of a 25-mm biotite flake that had been degraded for 18 months were removed with a razor blade and analyzed. There was 82 meq K/100 g in this edge material. This was much more K than Rausell-Colom *et al.* (1965) observed. The explanation probably lies in the large amount of edge expansion that occurred with particles in NaTPB solutions. As stated earlier, this amount of expansion could only occur if a wide separation of some of the layers occurred. This separation of layers would make it possible for K in solution or even KTPB to be present in this weathered zone.

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