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EXPANDABLE PHYLLOSILICATE REACTIONS WITH LITHIUM ON HEATING

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Abstract—A method using Li saturation and heating to 250°C to differentiate montmorillonite from beidellite and nontronite has been developed. The test utilizes three washings with 3 M LiCl and two washings with 0.01 M LiCl in 90% methanol to prevent dispersion. An 'infinitely thick' sample (6–8 mg/cm^2) on a glass slide is used to avoid the effects of the reaction of a thin clay film with sodium of the slide when it is heated at 250°C. Solvation with glycerol rather than ethylene glycol is used, because all of the Li smectites studied expanded to some extent in ethylene glycol after the heating. The smectites included several montmorillonites, a nontronite, and saponites. The presence of interstratified montmorillonite and beidellite layers was clearly shown by the test for several smectite samples, including the so-called beidellites from Beidell, Colorado, and Chen-yuan, Taiwan, and several soil clays. The test thereby provides more mineralogical information than the often-used arbitrary dividing point between montmorillonite and beidellite at 50% tetrahedral charge. Heating the Li-saturated clays at 250°C caused substitution of 35 to 125 meq/100 g of nonexchangeable Li. These amounts exceeded the changes in cation-exchange capacity plus Li by 4 to 21 meq/100 g, except for the end-member beidellite from the Black Jack mine, Idaho. Fusion with $LiNO_3$ at 300°C could not be used to differentiate between smectites instead of washing with LiCl solution and heating to 250°C, because fused montmorillonite subsequently expanded to 18 Å with glycerol. Large increases in nonexchangeable Li were caused by the fusion of smectites, a vermiculite, and two partially expanded micas.

Key Words—Beidellite, Lithium saturation, Montmorillonite, Nontronite, Soil clay, X-ray powder diffraction.

INTRODUCTION

The loss of expansion properties of montmorillonite samples after they were saturated with Li and Mg and heated at 100°–900°C was studied by González García (1950) and Hofmann and Klemen (1950); this property was contrasted with that of other smectites. Montmorillonites have also been heated at 300°C (Greene-Kelley, 1952), and 200°C (Greene-Kelley, 1953) and then solvated with glycerol for an X-ray powder diffraction (XRD) test of nonexpandability. Various systems have been used to test the expandability of heated montmorillonite, such as water saturation at various vapor pressures and solvation with glycerol or ethylene glycol (Brown, 1961). Anderson and Reynolds (1966) found that the Umiat, Alaska, bentonite which had been electrodialyzed did not qualify as a montmorillonite without subsequent drying with an excess of LiCl solution. In general, the saturation with Li has been accomplished by washings with LiCl solution and then washings with alcohol of various concentrations to remove the excess salt. During alcohol washings, the dispersion and hydrolysis of the Li-saturated clay has been a problem.

Inasmuch as no reliable procedure has emerged based on these and other experiments, more study of the problem was urged at the 1975 International Clay Conference (Bystrom-Brusewitz, 1976). Reliable tests for differentiating each smectite species are needed, especially because the AIPEA nomenclature committee (Bailey, 1980) recommended that smectite be accepted as a group name for phyllosilicates with layer charge between 0.2 and 0.6 per $O_{10}(OH)_2$ unit, in lieu of "montmorillonite-saponite."

The objective of this paper is therefore to report improvements in the criteria which differentiate montmorillonite from other smectites. The study was based on comparisons of classical specimens (Table 1) and their thermal reactions with Li.

EXPERIMENTAL

Samples of smectites, soils, vermiculite, and mica (Table 1) were dispersed, fractionated, and deferrated by conventional procedures (Jackson, 1979). Then 50 mg of clay was transferred into a 15-ml test tube and washed 3 times with aqueous 3 M LiCl and 2 times with 0.01 M LiCl in 90% methanol. A slurry was prepared with water and spread over 6.5 cm^2 on a petrographic microscope slide. The clay was allowed to dry slowly at 25°C and then heated overnight at 250°C in a muffle furnace. Such samples are hereinafter designated as "Li-250°C" clay. Curling of the clay film during heating, if it occurred, was prevented by wrapping thin bands of aluminum foil tightly around the edges of the clay-coated slide prior to heating (Brindley and Ertem, 1971). The Li-250°C clay slide was cooled in a desiccator, placed in a sealed glass container containing

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Table 1. Samples analyzed.

Materials	Reference or source
Bentonite, Upton, Wyoming ("Volcay")	American Colloid Company, Chicago, Illinois
Bentonite, Colony, Wyoming ("Blue")	Weaver *et al.* (1976)
Bentonite, Umiat, Alaska	Anderson and Reynolds (1966)
Montmorillonte ("Cheto"), Apache County, Arizona (SAz-1)	Source Clays Repository of The Clay Minerals Society
Nontronite, Grant County, Washington (SWa-1, "ferruginous smectite")	Source Clays Repository of The Clay Minerals Society
Saponite, Woodson County, Kansas	Jackson and Sridhar (1974)
Saponite, Vallejo, California	American Cyanamide quarry, courtesy J. L. Post
Beidellite, Beidell, Colorado	Larson and Wherry (1925), U.S. Geological Survey, Washington, D.C.
Beidellite, Black Jack mine, Idaho	Weir and Greene-Kelly (1962), National Museum of Natural History, Washington, D.C.
Beidellite, Chen-yuan, Taiwan	Chen *et al.* (1976), courtesy P. H. Hsu
Vermiculite, Grouse Creek, Utah	Abdel-Kadar *et al.* (1978)
Mica, Cornwall, United Kingdom[1]	English Mica Company, courtesy J. M. Huber Corporation, Macon, Georgia
Illite, Fithian, Illinois	Ward's Natural Science Establishment, Rochester, New York
Soil, Mollisol, Grand Forks, North Dakota	Lim and Jackson (1984)
Soil, Mollisol, Edmonton, Alberta	Lim and Jackson (1984)
Soil, Inceptisol, Overlander site	Lim and Jackson (1984)
Soil, Alfisol, Columbia, Missouri	20 km east of Columbia, Missouri, Wise farm

[1] Clear white mica ('micro mica') ground to pass 130-μm openings.

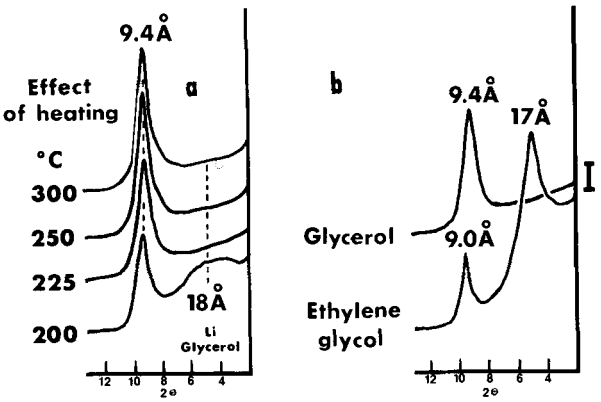


Figure 1. X-ray powder diffractograms of Li-saturated montmorillonite, from Upton, Wyoming, heated to various temperatures, illustrating (a) the irreversible collapse to a 9.4-Å basal spacing of the clay heated to 250°C with 16-hr glycerol solvation and (b) extensive expansion of the Li-250°C clay with ethylene glycol. CuKα radiation.

glycerol for solvation, and heated in an oven at 90°C for 16 hr. The clay was then examined by XRD by Ni-filtered CuKα radiation at a scanning speed of 2°2θ/min.

The following procedures were examined to determine the most effective method of testing: (1) heating at 250°C was compared with heating at 200°, 225°, and 300°C; (2) solvation of the Li-250°C clay with ethylene glycol (vapor phase, at 65°C) was compared with solvation with glycerol at various glycerol solvation times; (3) extraction of Na from an ordinary petrographic microscope slide by Li-250°C montmorillonite was measured; (4) the effect of different amounts of Li-250°C montmorillonite on the slide was determined; (5) various mixed-layer smectites were examined; and (6) the amount of Li fixed in nonexchangeable form by heating was compared to changes in cation-exchange capacity and expandability by glycerol.

The calcium cation-exchange capacity (CaEC) was determined by washing a sample first with 0.25 and 0.005 M $CaCl_2$ solutions and then with 0.25 M $MgCl_2$ solution to replace the Ca for atomic absorption measurement. The CaEC (meq/100 g) was obtained as the displaced Ca corrected by subtraction of the trace of Ca in the final washing solution determined by the weighing method of Jackson (1979). The Li-250°C clay was triturated with a glass rod to ensure complete dispersion. The total Li and K contents of the samples were measured by NH_4^+-saturation, digestion in HF-H_2SO_4, and flame emission spectrophotometry. Total Na was similarly determined on some samples. All determinations were made in duplicate; the means are reported on the weight basis after the samples were dried at 110°C.

RESULTS AND DISCUSSION

Selection of experimental criteria for the Li-250°C clay

Li-saturated montmorillonite (Upton, Wyoming) remained collapsed in part after being heated at 200°C and fully collapsed after being heated to 250°C (Figure 1a). The intensity of its 9.4-Å basal XRD reflections after glycerol solvation was greatest at 250°C; hence, this temperature was selected for further thermal treatments. The nature of the polar solvate, i.e., glycerol (Greene-Kelly, 1952) or ethylene glycol (Bystrom-Brusewitz, 1976), affected the expandability of the montmorillonite (Figure 1b). Irreversible collapse of the Li-250°C montmorillonite to a 9.4-Å spacing was

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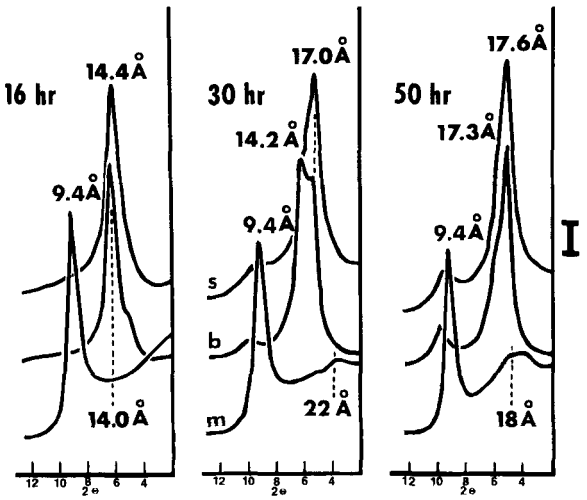


Figure 2. Expansion of Li-250°C smectites after glycerol solvation for 16, 30, and 50 hr (s = saponite, Kansas; b = beidellite, Black Jack mine, Idaho; m = montmorillonite, Upton, Wyoming). CuKα radiation.

obtained by glycerol solvation, as required for the test. Ethylene glycol largely re-expanded the Li-250°C montmorillonite to a 17-Å spacing and therefore could not be used for the test. These results agree with the order of penetration of polar liquid into heated minerals, i.e., ethylene glycol > glycerol > water (Johns and Tettenhorst, 1959; Calvet and Prost, 1971).

The duration of glycerol solvation in a closed container affected the physical swelling of the Li-250°C smectites. No re-expansion of the collapsed montmorillonite layers was observed within 16 hr of solvation (Figure 2). Beidellite (Idaho) and saponite (Kansas) gave broad XRD reflections at 14.0 and 14.4 Å, respectively. With increased solvation time (30–50 hr), the montmorillonite partially re-expanded to 18–22 Å. The XRD reflections of the beidellite and saponite shifted to 14.2–17 Å. The basal spacing of vermiculite, in contrast, remained at 14.2 Å throughout the experiment (data not shown). Therefore, the XRD powder pattern of Li-250°C smectites obtained after 16 hr (as contrasted 30–50 hr) of glycerol solvation was used as the standard montmorillonite test.

Chemical interaction between montmorillonites and the glass slides released a total of 66–83 meq Na/100 g of the Li-250°C clays compared with the negligible Na released from a slide coated with LiCl solution and heated to 250°C (Table 2). The NH_4-saturated montmorillonite controls which were not heated took up 38–39 meq Na/100 g. An 18-Å XRD reflection was produced by a glycerol-solvated, Li-250°C montmorillonite with sample coverage of 1.0 mg clay/cm² on the glass slide (Figure 3). In the Na-Li montmorillonite system, partial swelling of heated montmorillonite by the sorption of polar molecules occurred if the exchangeable Na was greater than 30% of the exchange

Table 2. Sodium released to clays coating plain microscope slides.

Sample	Slide treatment	Total Na (meq/100 g)
Montmorillonite, Colony, Wyoming	Li-250°C clay[1]	66.0
	Control NH_4^+-clay[2]	39.2
Montmorillonite, Upton, Wyoming	Li-250°C clay[1]	82.7
	Control NH_4^+-clay[2]	37.7
Control slide	With LiCl salt (heated)	0.01

[1] All slides clay-coated with 0.9 mg clay/cm². Li-250°C indicates the standard heating.
[2] The NH_4^+-clay slide was unheated.

capacity (Greene-Kelly, 1955; Calvet and Prost, 1971). To solve this problem, an 'infinitely thick' sample (6–8 mg clay/cm²) was prepared on a glass slide for thermal treatment at 250°C (Figure 3). Bystrom-Brusewitz (1976) suggested the use of a silica slide for this test; however, a weak 17-Å XRD reflection still was evident, probably because ethylene glycol was used for solvation in that study.

Several glycerol-solvated (16 hr), Li-250°C montmorillonites, including that from Umiat, Alaska, gave sharp 9.4-Å peaks, indicative of the desired irreversible interlayer collapse (Figure 4). The results for the Umiat clay contrast with to those of Anderson and Reynolds (1966). The exchange resin used by them incompletely converted the Umiat H clay into a Li clay, inasmuch as some Al saturation was shown by their data.

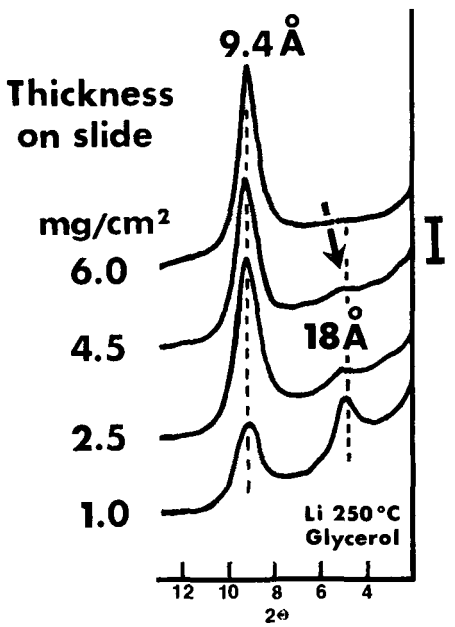


Figure 3. Effect of the amount of montmorillonite (Upton, Wyoming) on the X-ray powder diffraction intensity. Arrow indicates the effect of clay interaction with Na of the slide. CuKα radiation.

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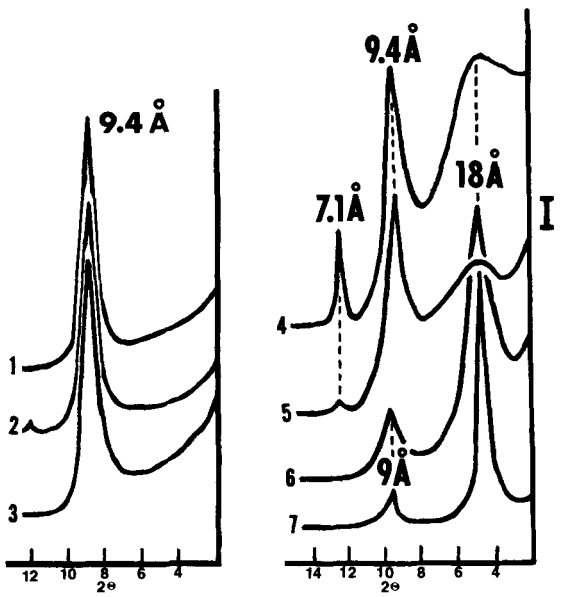


Figure 4. X-ray powder diffractograms of Li-250°C smectites solvated with glycerol for 16 hr illustrating the irreversible collapse of montmorillonites (1 = Colony, Wyoming; 2 = Umiat, Alaska; 3 = Apache County, Arizona) and the partial or complete expansion of other smectites (4 = beidellitic kaolinitic montmorillonite, Taiwan; 5 = beidellitic montmorillonite, Colorado; 6 = nontronite, SWa-1, ferruginous smectite, Washington; 7 = saponite, California). CuKα radiation.

The re-expansion of nontronite and saponite to 18.0 Å (Figure 4) is in keeping with their tetrahedral charge. The results with this nontronite contrast with those with an iron-rich montmorillonite (Wildman *et al.*, 1968). The beidellite from the Black Jack mine, Idaho, appears to be a unique, high-charge end-member specimen of the mineral (Weir and Greene-Kelly, 1962). Its unusually high charge probably accounts for the longer solvation time of 50 hr needed (Figure 2) to expand its basal spacing to 17.3 Å than the 16 hr needed for the expansion of nontronite and saponite (Figure 4).

End-member smectites are rare (Glaeser *et al.*, 1967); most smectites exhibit charge heterogeneity among their layers (Tettenhorst and Johns, 1966; Lagaly and Weiss, 1976). The samples from Beidell, Colorado (Larsen and Wherry, 1925) and Chang-yuan, Taiwan (Chen *et al.*, 1976) were found to be montmorillonite-rich (9.4 Å) with some interstratified beidellite (broad 18-Å reflections, Figure 4). Both octahedral and tetrahedral charges were also indicated in the soil clays (Figure 5a). The fine clay fractions of the Mollisol C horizons (Figure 5a) contained both montmorillonite (the 9.4-Å reflection was combined with the broadened 10-Å mica reflection) and interstratified beidellite (broad 18-Å reflection). The soils were derived from glacio-lacustrine sediments mainly of Cretaceous age and rich in smectite (sharp 18-Å reflections, with Mg-glycerol; Lim and Jackson, 1984); the beidellite has been attributed to mica weathering. The clay of the Inceptisol developed in loess at the Overlander, Alberta, site is composed of mica, vermiculite, and montmorillonite (Figure 5a).

The Putnam B clay, which Marshall *et al.* (1942) considered to be mainly beidellite, appears to be a mixed-layer dioctahedral smectite. Its content of montmorillonite is indicated by the broadened 9.4-Å reflection (Figure 5a, curve 4). Its considerable beidellite content is indicated by the mixed-layer XRD reflection in the region 11–20 Å. A mixed-layer "beidellitic montmorillonite" from New South Wales has been reported (Hamilton, 1971), similar to the Colorado and Taiwan samples (see above). The Putnam clay, by analogy, could be termed a "montmorillonitic beidellite." The XRD patterns of the Li-250°C clays provide clear indications of their degree of mixed-layering (Figures 4 and 5a). This XRD characterization of mixed-layering seems preferable to a singular delineation of montmorillonite vs. beidellite (Ross and Mortland, 1966) at the mid-point of tetrahedral-octahedral origin of charge.

Effect of heating Li-saturated smectites on CaEC and Li content

In a preliminary experiment in which Li-saturated clays were heated at 250°C, an appreciable loss of clay during the methanol-acetone washings was indicated by lower CaEC values (compared to those with 0.01 M LiCl washings) determined after the heating but before the total Li determinations (Table 3). Also, low CaEC values and large contents of nonexchangeable Li were observed. In further work with 0.01 M LiCl washings and 250°C heating, a 70–80% decrease in the CEC of montmorillonite (Table 4) accompanied loss of the interlayer swelling in glycerol (Figure 4). In contrast, about 70% of the CaEC of the Idaho beidellite and nontronite and nearly 85% of the CaEC of the saponites remained. The fact that only 30–50% of the CaEC of the Colorado and Taiwan samples remained (Table 4) was in keeping with their high contents of montmorillonite interstratified with the beidellite (Figure 4b).

The nonexchangeable Li after the Li-250°C treatment paralleled the decrease in CaEC; however, the sum of the Li increase plus the remaining CaEC exceeded the CaEC of the untreated smectites by 1–21 meq/100 g (Table 4). Inasmuch as the total Li was determined after the extensive washings of the sample to determine CaEC, the Li must have been tightly incorporated in the clay, having migrated into vacant octahedral sites (Hofmann and Klemen, 1950; Heller-Kallai and Rozenson, 1980) rather than having been retained in the mica-like hexagonal cavities adjacent to the interlayers (González García, 1950; Tettenhorst, 1962; Calvet and Prost, 1971). This conclusion is supported by the fact that the nontronite, saponites, and

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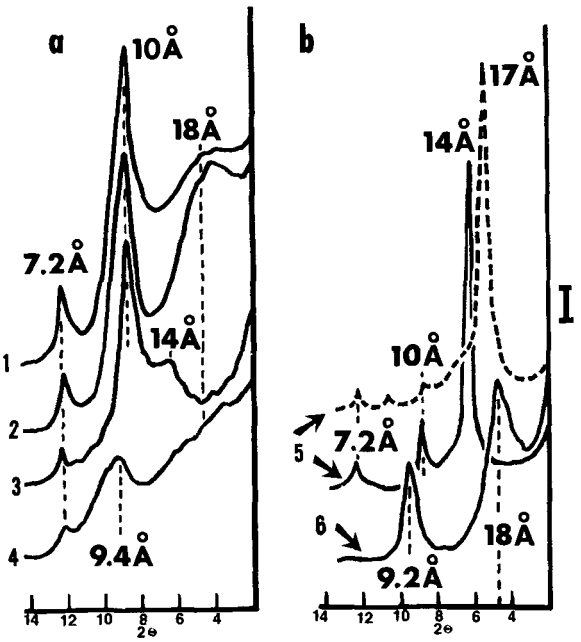


Figure 5. X-ray powder diffractograms (a) of Li-250°C soil clays (1 = Mollisol C, Grand Forks, North Dakota; 2 = Mollisol C, Edmonton, Alberta; 3 = Inceptisol C, Overlander, Alberta; and 4 = Putnam B, Columbia, Missouri) and (b) clays after fusion in $LiNO_3$ at 300°C (5 = mica expanded to 14 Å with glycerol and at 17 Å with ethylene glycol; 6 = extensive expansion with glycerol of montmorillonite, Upton, Wyoming, after the fusion). CuKα radiation.

beidellites readily expanded after being heated to 250°C. The Idaho beidellite showed only 0.7 meq/100 g gain in the sum of Li + CaEC, and therefore the amount of incorporation of Li almost equalled the CaEC decrease (Table 4). The excess sum of Li increase + CaEC

Table 3. Cation-exchange capacity[1] and Li content of Li-smectites prepared by different methods and heated to 250°C.

Sample	Treatment after three washings with 3 M LiCl	CaEC (meq/100 g)	Total Li (meq/100 g)
Montmorillonite, Arizona	0.01 M LiCl[2]	17.5	126.4
	+ methanol[3]	15.0	119.4
Nontronite, Washington	0.01 M LiCl	79.6	46.4
	+ methanol	76.4	45.6
Saponite, California	0.01 M LiCl	94.9	26.7
	+ methanol	91.7	22.6

[1] Measured with Ca (CaEC) after the heating of the Li-saturated samples.
[2] Two washings in 0.01 N LiCl in 90% methanol.
[3] One washing with salt-free 80% methanol, 5 washings with 99% methanol, and 2 washings with acetone.

in the other samples may have occurred through displacement of structural protons by Li (Farmer and Russell, 1967; Ertem, 1972; Heller-Kallai, 1976).

Smectites have been reported to fix alkali elements and to collapse to a 10-Å basal spacing on exposure to mild diagenesis (Weaver, 1958). Fusion in $LiNO_3$ (for 24 hr at 300°C, 1:60 clay: flux ratio) will reverse this process and release interlayer K from micas (White, 1956). As reviewed by Douglas (1982), $LiNO_3$ fusion has sometimes been substituted for 3 M LiCl washings in the test for beidellite vs. montmorillonite. The introduction of Li by this fusion was therefore compared to that with LiCl washings and heating at 250°C. Fused montmorillonite failed the test of retention of a 9.4-Å spacing with glycerol (Figure 2), because it expanded to 18 Å (Figure 5b).

The nonexchangeable Li after the fusion was almost equal to the CaEC of the montmorillonite and non-

Table 4. Effect of heating Li-saturated clays[1] on cation-exchange capacity[2] and Li content of smectites.

Sample	Untreated clay		Li-250°C clay		
	CaEC (meq/100 g)	Total Li (meq/100 g)	CaEC (meq/100 g)	Total Li (meq/100 g)	Increased sum[3] (meq/100 g)
Montmorillonite					
Arizona	132.1	3.0	17.5	126.4	8.8
Alaska	94.3	0.3	17.9	84.5	13.8
Wyoming (Upton)	90.8	0.1	21.1	76.5	6.7
Beidellite					
Idaho	118.7	0.0	85.0	34.4	0.7
Colorado	83.7	0.1	42.2	62.3	20.5
Taiwan	96.1	0.0	30.2	69.8	3.9
Nontronite					
Washington	109.7	0.0	79.6	46.4	16.3
Saponite					
Kansas	108.0	2.7	89.9	33.7	12.9
California	112.8	0.2	94.9	26.7	8.8

[1] Saturated by washings with 3 M LiCl solution.
[2] Measured with Ca (CaEC) before and after heating Li clays at 250°C.
[3] The sum (CaEC + total Li) from which the CaEC of the untreated sample has been subtracted.

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Table 5. Effect of $LiNO_3$ fusion at 300°C on cation-exchange capacity[1] and K and Li contents of 2:1 phyllosilicates.

Sample[2]	Untreated clay		$LiNO_3$ fused clay		
	CaEC (meq/100 g)	Total K (meq/100 g)	CaEC (meq/100 g)	Total K (meq/100 g)	Total Li (meq/100 g)
Montmorillonite, Arizona	132.1	0.8 (3.0)[3]	32.4	0.5	129.6
Nontronite, Washington	109.7	0.6 (0.0)	111.5	0.3	105.0
Vermiculite, Utah	154.2	12.1 (1.1)	153.4	4.9	53.6
Illite, Illinois	27.0	130.7 (0.5)	86.0	51.6	63.6
Mica (silt), United Kingdom	2.4	197.9 (9.4)	30.2	154.4	36.1

[1] Measured with Ca (CaEC), before and after fusion in $LiNO_3$.
[2] Clay size (<2 μm), except for mica silt (2–5 μm) separated from "micro mica," a clear white finely ground mica (Table 1).
[3] Numbers in parentheses are the Li contents of the original samples.

tronite and was one-third of that of the trioctahedral vermiculite (Table 5). The fact that the smectites and vermiculite retained their original CEC indicates that the $LiNO_3$ fusion caused more extensive structural changes than were noted for the Li-250°C clays (Table 4). The trioctahedral vermiculite retained its XRD reflection at 14 Å (data not presented).

Dioctahedral illite and mica that were fused in $LiNO_3$ showed CaEC gains of 59.0 and 27.8 meq/100 g, respectively, values that are nearly equivalent to 63.1 and 26.7 meq Li/100 g gained (Table 5). This equivalence corresponds to the near identity of the decrease in CaEC with the increase of Li in the end-member Idaho beidellite (Table 4). Some structural changes in the micas during the $LiNO_3$ fusion are, however, indicated by the excess of K released over the gain in CaEC (20.1 and 15.7 meq/100 g, respectively, Table 5).

The CaEC values of the opened micas, calculated by excluding the remaining mica (based on K remaining, Table 5) were about 120 to 140 meq/100 g. The slow opening beyond 14 Å of the high-CaEC Idaho beidellite (Table 4) with glycerol (30–50 hr, Figure 2) corresponds to the 14-Å XRD response to glycerol for the highly charged, opened part of the micas (Figure 5b), comparable to the expansion of vermiculite (Barshad, 1954; Alexiades and Jackson, 1965).

CONCLUSIONS

Heating Li-saturated montmorillonites to 250°C overnight (Li-250°C clay) allowed them to be differentiated from all other smectites by their resistance to solvation with glycerol vapor for 16 hr at 90°C (Figures 2 and 4). Lower temperatures of heating (Figure 1) or substitution of ethylene glycol for glycerol did not yield a satisfactory result (Figure 1). The effect of Na extraction from the glass slide by the Li-clay can be nullified by the use of 6 mg/cm² of clay (Figure 3). Fusion in $LiNO_3$ cannot be substituted for LiCl washings for a satisfactory montmorillonite test (Figure 5b).

Smectitic clays from rocks and soils are commonly mixtures of montmorillonite and other clays with some tetrahedral Al sheets that allow the corresponding layers to open with the Li-250°C test (Figures 4 and 5). The nonexchangeable Li of the dioctahedral beidellite and micas is equivalent to change of layer charge. In contrast, the nonexchangeable Li is nonstoichiometric with the change in layer charge of nontronite, montmorillonite, trioctahedral vermiculite, and saponite (Tables 4 and 5), which is interpreted as indicating structural changes.

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