

## SAPONITE FROM NEAR BALLARAT, CALIFORNIA

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**Abstract**—White saponite occurs in joints and open fracture zones in metamorphosed dolomitic limestone near Ballarat, California. The saponite appears to have formed by hydrothermal alteration, possibly during Pliocene times. The material shows a 06/ X-ray powder diffraction peak at 1.529 Å, Al<sub>2</sub>O<sub>3</sub> and MgO contents of about 4.4 and 23.5%, respectively, and a half-cell octahedral atoms summation value of about 2.82. The saponite appears to consist of a single, uniform clay species; the main impurities are fine shards of diopside and tremolite. The infrared and thermal properties of the Ballarat saponite are similar to those of the Allt Ribhein saponite. It has a lower water-holding capacity than montmorillonite and is characterized by lower Atterberg limits and expansion pressures and higher compaction densities. The apparent density of the saponite, 2.865 g/cm<sup>3</sup>, is greater than that of montmorillonite. This saponite is available from the Source Clays Repository of The Clay Minerals Society.

**Key Words**—Engineering properties, Hydrothermal alteration, Saponite, Sepiolite, Source clay, X-ray powder diffraction.

### INTRODUCTION

While investigating a deposit of white diopside in Panamint Valley near Ballarat, Inyo County, California, adjacent bodies of white clay were observed. The material in a nearby clay pit was moist at shallow depth under extremely hot, dry conditions. The purpose of this investigation is to describe the properties of this clay, including its physical, chemical, and engineering features. The clay was described as saponite by Post (1981). Representative quantities of this clay has been added to the Source Clays Repository of The Clay Minerals Society.

### LOCATION AND GEOLOGY

The saponite is found about 3 miles northeast of Ballarat (Figure 1) in Sec. 26, T21S, R44E, on a steep hillside at an elevation of about 1600 ft (488 m) in South Panamint Valley. Ballarat is an abandoned gold mining center near the base of the west flank of the Panamint Range on the east side of South Panamint Playa at an elevation of about 1060 ft (323 m) in the lower Sonoran desert physiographic region (U.S.G.S. Telescope Peak Quadrangle topographic map, scale 1:62,500, 1952). Dual units are given on Figure 1 for use in conjunction with the topographic map. The geology of the area was first described by Murphy (1932); a more detailed study of the area was made by Labotka (1978). The remnants of Pleistocene Lake Panamint, which covered the clay deposits, were investigated by Smith (1976), and the clay mineral content of the bed of South Panamint Playa was discussed by Droste (1961).

The saponite occurs in joints and open fracture zones in metamorphosed dolomitic limestone in the Late Cambrian Noonday Dolomite Formation near the base

of the Panamint Range. The highly brecciated and crumpled limestone is light brown to buff on weathered surfaces and gray where fresh. Lenses of bladed tremolite are common. The saponite occurs chiefly in bedding plane joints, and thin, fibrous sheets of sepiolite locally occur against the upper fracture and joint surfaces. The limestone structure containing the clay minerals was submerged in Lake Panamint at least five times in the last 100,000 years (Smith, 1976), and during evaporation of the lake the limestone was subjected to surface erosion. South Panamint playa, two miles to the west, is a bolson structure; hence, all erosion products in the watershed are carried toward the lake bed. The clays in the lake bed are montmorillonite, illite, chlorite, and kaolinite (Droste, 1961); magnesium salts are extremely rare.

The larger deposits of saponite are in white, surface halo zones of limestone that appear to have been leached by hydrothermal action. The clay pit is moist; there is a flowing warm spring near the deposit. There appears to have been hydrothermal replacement of metamorphosed dolomitic limestone below the weathering profile during the numerous, recent faulting and flexing episodes in the area. If the Mg source for the saponite and sepiolite were the dolomite, the alteration products should not be limited to the fracture zones but also found in the nearby playa. Magnesian clays and salts are extremely rare in the playa sediments, hence, the Mg must have been supplied by phreatic ground water.

Thin sheets of sepiolite appear to have formed at the expense of the saponite in the lake during its periods of evaporation and increased salinity between Plio-Pleistocene glacial episodes. Some flexible sheets extend into the saponite bodies. The saponite may have formed earlier, possibly in Pliocene times during pe-

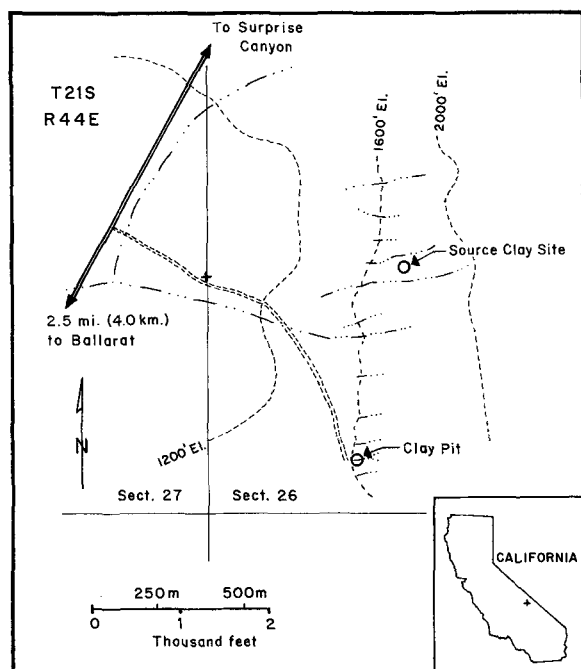


Figure 1. Source Clay site and clay pit site, in relation to Ballarat, California, and Sect. 26, T21S, R44E. The  $\frac{1}{4}$  corner for Sect. 26/27 is visible from the trail to the pit.

riods of increased tectonic displacement. Relatively little tectonic displacement occurred during the existence of Panamint Lake (Smith, 1976). The formation of the sepiolite is described elsewhere (Post and Janke, 1983).

The dominant minerals found randomly distributed throughout the saponite are shards of tremolite and diopside; lesser amounts of calcite, quartz, feldspar, and talc fragments are also present, with traces of zircon. The evaporite minerals present are chiefly halite, gypsum, and thenardite and tend to be concentrated near the surface. Kerolite was found in surface samples taken near the clay pit. The clay seams tend to be lenticular and generally not more than 0.3 m thick. The Source Clay material contains about 70% saponite and the clay pit material about 55% saponite, along with the impurities listed above.

#### ANALYTICAL PROCEDURE

Specimens of the Source Clay saponite were used for all tests conducted except for comparison of chemical analyses, where the pit clay also was used. Physical tests, such as Atterberg limits, were performed using air-dried, pulverized whole samples, and chemical and X-ray powder diffraction (XRD) analyses were obtained from clay separated by water suspension.

The clay-size fraction was separated by using a blender to disperse about 60 g of saponite with 3 g of

Calgon in water. Immediately after mixing, the slurry suspension was placed in a standard 1000-ml sedimentation cylinder, upended to complete agitation of the slurry, then allowed to settle for 5 hr after which the upper half of the suspension, containing  $<2\text{-}\mu\text{m}$  size particles, was removed by siphoning. XRD scans of the dried residue showed saponite and about 4% halite. The halite, and any residual Calgon, were removed by successive decantation.

XRD patterns were made using a Picker X-ray diffractometer with a  $1^\circ$  divergence slit and a  $1^\circ$  scatter slit in conjunction with a 0.002 in. receiving aperture. Ni-filtered  $\text{CuK}\alpha$  radiation was used with a scan rate of  $1^\circ/2\theta/\text{min}$ . Differential thermal analyses were made between  $25^\circ\text{C}$  and  $1120^\circ\text{C}$  using a Perkin-Elmer DTA 1700 thermal analyzer with a heating rate of  $20^\circ\text{C}/\text{min}$  using an alundum reference material and a nitrogen purge. Infrared (IR) absorption spectra were obtained using KBr discs with a Perkin-Elmer 337 grating IR spectrophotometer having a light frequency response varying from  $4000$  to  $400\text{ cm}^{-1}$ .

The chemical compositions of the two Ballarat saponite specimens and a specimen of saponite from American Canyon were determined by X-ray fluorescence (XRF) spectrographic analysis. The results were adjusted for water content on the basis of gravimetric loss of the specimens when heated. The Picker dual-function spectrodiffractometer used for XRF analyses was described by Ashby *et al.* (1963). The Al contents were verified using a TAP analyzing crystal for greater accuracy. Na, F, and Li were determined by atomic absorption spectrophotometric methods.

#### RESULTS

XRD patterns show that two different types of saponite are present in most of the clay samples investigated, most specimens giving two clearly separated basal spacings. The Na-salts, mainly halite and thenardite, were mostly removed when specimens of clay were soaked prior to wet sieving, leading to a reduction in intensity of the Na-saponite basal peaks and increase in intensity of the Ca-saponite basal peaks without benefit of ion exchange. The reason for this unusual behavior is not known. The XRD pattern for the Ca-saponite is given in Table 1, along with the Na-saponite pattern produced when Calgon was used as a dispersant in the clay-water suspensions. The diffraction indices were assigned as suggested by Faust *et al.* (1953) and Suquet *et al.* (1975). The first-order basal spacings,  $15.15\text{ \AA}$  for the Ca-saponite and  $12.55\text{ \AA}$  for the Na-saponite, were shifted to form a very strong sharp peak at  $17.0\text{ \AA}$  when the saponite was solvated with ethylene glycol. The higher orders of the basal spacings are exact multiples of the expanded first-order basal reflection. The relative intensity of the first five basal reflections is given in Table 1, and the remaining reflections are: (006) m, (008) w, (009) vw, with no (007) or (0010)

Table 1. X-ray powder diffraction data for Ballarat saponite.

Indices <sup>1</sup>	Air-dry				Ethylene glycol solvation	
	Ca <sup>2+</sup> d (Å)	Sat. I	Na <sup>+</sup> d (Å)	Sat. I	d (Å)	I
001	15.15	vvs	12.55	vvs	17.0	vvs
002	—	—	—	—	8.50	s
003	5.05	w	—	—	5.65	m
11; 02l	4.575	mw	—	—	—	—
004	3.79	vw	3.134	m	4.250	w
005	3.03	w	—	—	3.400	s
13l	2.60	mw	—	—	—	—
20l	2.52	mw	—	—	—	—
22; 04l	2.30	vw	—	—	—	—
21; 15l	1.73	w	—	—	—	—
06; 33l	1.529	mw	—	—	—	—
26l	1.32	w	—	—	—	—
40l	1.28	vw	—	—	—	—

<sup>1</sup> The Miller indices are assigned according to the data given by Faust and Murata (1953) and Suquet *et al.* (1975). The hk reflections for the Na-saponite are essentially the same as for the Ca-saponite.

reflections. The hk/ spacings of more than 50 samples collected across about 5 km of exposed fracture zones in the limestone do not vary, indicating that the same saponite clay is present throughout the area.

When the saponite was heated, the 14-Å peak slowly shifted to a smaller spacing while retaining its intensity. At about 600°C the peak, with a spacing of 12.25 Å, began to collapse and shifted to 9.85 Å. When cooled from 650°C in open air the basal peak readily rehydrated to 14 Å in a few minutes, similar to the Allt Ribhein saponite described by Mackenzie (1957). When heated above 750°C, the clay crystal structure was destroyed. The hk reflections, other than the 02 and 06 reflections are very broad as shown in Figure 2.

The positions of the IR absorption bands for the air-dried, untreated Ballarat saponite (Table 2) are very similar to those of the Allt Ribhein saponite (Farmer, 1958). The chemical composition and structural formulae of two saponite specimens are given in Table 3. One specimen was from the site of the Source Clay and the other from the clay pit to the south (Figure 1). No differentiation of ferrous and ferric iron was made. A saponite specimen from the clay pit also contained 97 ppm Li and 242 ppm F. The saponite analyses given by Weaver and Pollard (1973) indicate that although most of the Al content of the saponite is contained in the tetrahedral layer the octahedral Al half-cell content ranges from 0.00 to 0.27 of the octahedral positions filled, with most samples having less than 0.05 positions occupied by Al. The chemical analysis and structural formula of a more typical saponite, from American Canyon, California, is included in Table 3 for comparison. This saponite was investigated by Ab-

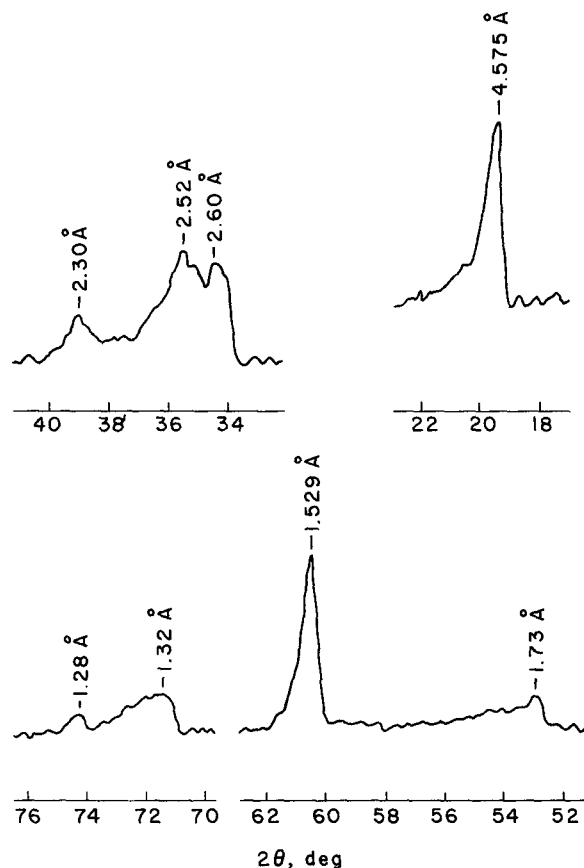


Figure 2. X-ray powder diffractometer trace for Ballarat saponite, for segments from 18°2θ to 76°2θ showing the hk reflections, using a scan rate of 1°2θ and count rate of 400 cps, time constant of 3 sec, 1° divergence and scatter slits and 0.002 in. receiving aperture (CuKα radiation).

Table 2. Position of infrared absorption bands for saponite from Ballarat, California, and Allt Ribhein, Skye.

Frequency and relative degree of absorption		
Ballarat	Allt Ribhein	Mode <sup>1</sup>
(cm <sup>-1</sup> )	(cm <sup>-1</sup> )	
3702 m	3697 w	(OH) <sup>-1</sup> str.
3670 wm	3670 w	(OH) <sup>-</sup> str.
1065 s	1056 vs	Si-O <sub>v1</sub> ⊥
1010 vs	1005 vs	Si-O <sub>v3</sub>
805 vw	809 w	Al-O
695 w	692 m	Si-O <sub>v2</sub> ⊥
655 m	655 m	Si-O <sub>v4</sub>
530 m	534 m	Mg-O <sub>v6</sub> ⊥
460-445 s	464-450 s	Mg-O <sub>v7</sub>

Relative degree of absorption: s = strong; m = moderate; w = weak; str. = stretching.

<sup>1</sup> Farmer (1958).

Table 3. Chemical analyses and half-cell structural formulae of Ballarat and American Canyon saponites.

	Ballarat		American Canyon saponite
	Source Clay	Pit clay	
SiO <sub>2</sub>	51.26	50.25	43.64
Al <sub>2</sub> O <sub>3</sub>	4.42	4.44	5.72
Fe <sub>2</sub> O <sub>3</sub>	1.14	0.50	7.27
MgO	23.54	23.81	19.35
CaO	1.25	1.70	2.43
K <sub>2</sub> O	0.18	0.10	0.00
Na <sub>2</sub> O	1.14	0.76	0.04
TiO <sub>2</sub>	0.09	0.03	0.05
MnO	0.03	0.02	0.29
NiO	0.04	0.07	0.38
Cr <sub>2</sub> O <sub>3</sub>	—	—	0.22
H <sub>2</sub> O+	6.58	7.25	7.60
H <sub>2</sub> O-	10.08	10.76	12.67
	99.75	99.69	99.66
<b>Octahedral</b>			
Al	0.15	0.15	0.04
Fe	0.06	0.03	0.44
Mg	2.61	2.65	2.32
Σ	2.82	2.83	2.80
<b>Tetrahedral</b>			
Al	0.23	0.24	0.50
Si	3.77	3.76	3.50
<b>Interlayer</b>			
Ca/2	0.19	0.27	0.43
Na	0.16	0.11	0.00
K	0.02	0.01	0.00

The H<sub>2</sub>O values are calculated from specimen weight at 110°C.

del-Kader *et al.* (1978) and Post (1981), and was originally described by both as vermiculite. The Ballarat saponite, with 0.16 half-cell octahedral positions filled with Al, is comparable to the saponite from California described by Ross and Hendricks (1945), except for higher Si content of the Ballarat saponite.

The results of differential thermal analysis (DTA) and thermogravimetric analysis (TGA) of Ballarat saponite are shown in Figure 3. Two separate temperature scales are given because the DTA apparatus did not heat at a uniform rate at lower temperature. The DTA pattern closely resembles the DTA pattern given by Faust and Murata (1953) for a saponite from near Reno, Nevada. The Ballarat saponite has a strong endotherm at 148°C, and a weaker one at 810°C. Enstatite was formed when the saponite was fired to 1050°C. The weight change (Figure 3) is plotted on the basis of specimen weight at 300°C, as was done by Nutting (1943), for determining the constitutional water content and the interlayer water content. The dehydration curve was made by heating about 15 g of pure clay in a ceramic cup to equilibrium weight at selected temperature intervals and determining the weight each time.

The chemical analyses given in Table 3 were cal-

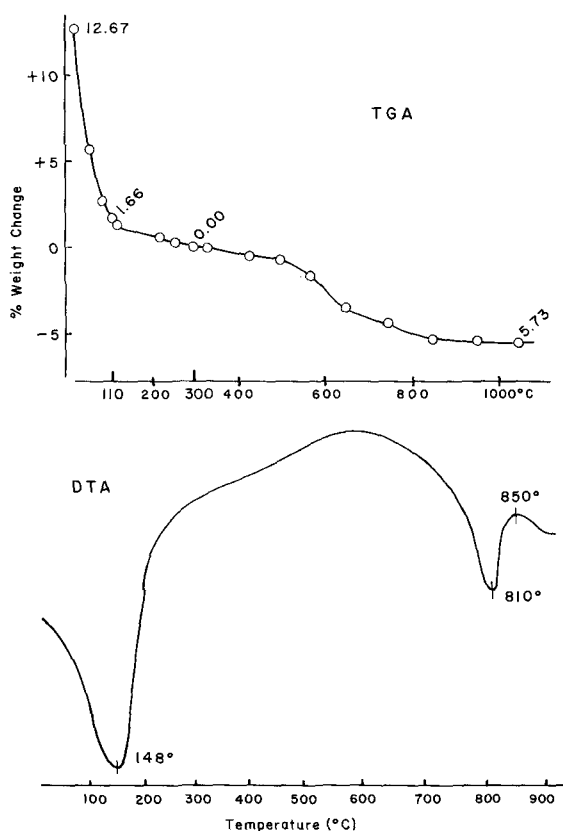


Figure 3. Differential thermal analysis and thermal gravimetric analysis patterns of the purified Ballarat saponite. The weight change upon heating is given with respect to 300°C equilibrium weight.

culated for H<sub>2</sub>O taken at 300°C because of the use of XRF analytical methods, then adjusted to H<sub>2</sub>O content at 110°C. The H<sub>2</sub>O contents for the temperature change from 110°C to 300°C are 1.52, 2.33, and 2.95% for the Source Clay, pit clay, and American Canyon clay, respectively. Mackenzie (1957) suggested that for saponite some of the H<sub>2</sub>O+ water, calculated on the basis of weight at 300°C, may be interlayer water. The water loss for the purified Source Clay saponite, when heated from 300°C to 1050°C, was 5.73%, and the calculated excess interlayer water was 1.12% at 300°C.

The untreated saponite has a cation-exchange capacity (CEC) of 73.8 meq/100 g (Ca/Mg) and 58.3 meq/100 g (K/NH<sub>4</sub>) as determined by a centrifuge washing method (Jackson, 1969; CEC determinations courtesy J. B. Dixon, Texas A&M University, College Station, Texas 77843). The sample contained a trace of calcite that may have contributed to the higher CEC value. The lower value may be due in part to fixation of K on oven drying during determination of the CEC by the K/NH<sub>4</sub> method. Thus, the presence of some high-charge layers is suggested. The two CEC values cate-

Table 4. Physical properties of unprocessed Ballarat saponite.

Engineering tests:		ASTM test method		
Atterberg limits	LL = 71 PI = 31			D 423-66 (1972) D 424-59 (1971)
Remolded density $\gamma_d$ =	1.821 g/cm <sup>3</sup>			D 1557-70
Optimum moisture =	18.0%			
Linear expansion	Ca <sup>2+</sup>	0.57 cm/cm	Na <sup>+</sup>	0.70 cm/cm
Expansion pressure		7.4 kg/cm <sup>2</sup>		16.8 kg/cm <sup>2</sup>
API drilling mud tests:		Saponite	Wyoming bentonite	
% less than 2 $\mu$ m		70	70	
pH (6% solids)		7.2	8-9	
Apparent viscosity (sec/qt)		9	>15	
Plastic viscosity (centipoises)		4	>8	
Yield point (lb/100 ft <sup>2</sup> )		10	<14	
Gel strengths (lb/100 ft <sup>2</sup> )		10-14	6-20	
Filtration (cm <sup>3</sup> /min)		16.2	13-15	

Includes the results of American Society for Testing and Materials (ASTM) test methods, and American Petroleum Institute (API) drilling mud tests.

gorize the cation-exchange characteristics of the Ballarat saponite sample, with its recognized carbonate and silicate impurities, by employing two different pairs of ions and procedures intended to permit free exchange (Ca/Mg) and to permit fixation (K/NH<sub>4</sub>). The material has an average clay-size content of 71%, as determined using Test Method ASTM D422-63. Assuming that all of the clay-size material is saponite and that all of the remaining material is composed of accessory minerals, the adjusted value of 104 meq/100 g (Ca/Mg test) for pure Ballarat saponite is comparable to the CEC value of 109 meq/100 g given for the Allt Ribhein saponite (Mackenzie, 1957).

The apparent density of the pure saponite averaged 2.865 g/cm<sup>3</sup>, using the ASTM D 854-58 Standard Test Method, in comparison to 2.790 g/cm<sup>3</sup> for Otay, California, bentonite (API 24) and 2.815 g/cm<sup>3</sup> for Santa Rita, New Mexico, bentonite (API 30). According to Deeds and van Olphen (1963), the apparent density in water of most expanding clays is equal to the crystallographic density of the material rather than its true density. They found the apparent density of Wyoming Na-bentonite to average 2.795 g/cm<sup>3</sup>.

Physical properties measured by standard American Society for Testing and Materials (ASTM) tests of the Ballarat saponite are given to relate its physical characteristics to those of montmorillonite (Table 4) because saponites are also encountered during highway construction and maintenance (Post, 1981). Also, when saponite is considered for use as drilling mud, the American Petroleum Institute (API) tests are required. The Atterberg limits of the saponite, which indicate the water-holding characteristics of the clay, are less than those of montmorillonite. Brown Porterville clay has a liquid limit (LL) of 113 and a plasticity index (PI) of 60 (Post and Janke, 1976) in comparison to a LL of 71 and a PI of 31 for the Ballarat saponite.

The remolded density of the unprocessed saponite, 1.821 g/cm<sup>3</sup>, is greater than that of remolded montmorillonite mainly because of the lower water-holding capacity of the former. In comparison, Otay bentonite, API 24, gives a remolded density of 1.365 g/cm<sup>3</sup>. The expansion volume and expansion pressure (with no volume change) of the Na-saponite are somewhat less than those values for montmorillonite (Post, 1981), and the Ca-saponite gives only about half as much expansion pressure as the Na-saponite. Porterville clay gave an expansion pressure of 22.4 kg/cm<sup>2</sup> in comparison to 16.8 kg/cm<sup>2</sup> for Na-saponite and 7.4 kg/cm<sup>2</sup> for Ca-saponite.

The drilling mud test data for unprocessed Ballarat saponite (Table 4), provided by W. J. Miles, Miles Industrial Minerals Research, Golden, Colorado 80401 (personal communication), indicate that in comparison to Belle Fourche, Wyoming, bentonite the saponite would be effective as a drilling mud, especially with its high-temperature stability.

The apparent viscosity was measured in sec/qt using a Marsh funnel, and the plastic viscosity was measured in centipoises using a Fann V-G meter. The yield point and gel strength were measured in lb/100 ft<sup>2</sup> also using the Fann V-G meter, and the filtration rate was measured in cm<sup>3</sup>/30 min using a filter press. The measurement units are peculiar to these testing instruments.

## CONCLUSION

Both of the Ballarat saponite specimens described here have essentially the same chemical composition. The regular order of the XRD basal spacings, the single sharp peak at 17.0 Å produced upon ethylene glycol solvation, and the decrease of the peak spacing to 9.85 Å when the saponite was heated to 650°C indicate that no mixed-layer structures are present. A comparison



of the physical characteristics of saponite with montmorillonite indicates that the two materials may be treated in the same manner during road construction and maintenance. The API test data suggest that this saponite may be useful as a drilling mud.

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(Received 4 April 1983; accepted 14 June 1983)

**Резюме**—Белый сапонит выступает в соединениях и открытых расщелинах в метаморфизованном доломитическом известняке вблизи Балларат в Калифорнии. Сапонит формировался, вероятно, путем гидротермального изменения, возможно в течение плиоценовой эпохи. Материал показывает дифракционную линию 061 при 1,529 Å, содержания  $Al_2O_3$  и  $MgO$  разны 4,4 и 23,5%, соответственно, и суммарное значение октаэдрических атомов равно 2,82. Кажется, что сапонит состоит из простых, однородных глинистых веществ; главными включениями являются тонкие осколки диоксида и тремолита. Инфракрасные и термические свойства сапонита из Балларат являются похожими на свойства сапонита из Аллт Рибхайн. Этот материал характеризуется меньшей способностью удерживания воды чем монтмориллонит и более низкими границами Аттерберга и давлениями экспансии и более высокими плотностями компакти. Кажущаяся плотность сапонита,  $2,8659/cm^3$  является большой, чем значение для монтмориллонита. Этот сапонит доступен со Склада Источниковых Глин Общества Глинистых Минералов. [E.G.]

**Resümee**—Weißer Saponit tritt in Klüften und offenen Spalten im metamorph beeinflussten dolomitischen Kalkstein nahe Ballarat, Kalifornien, auf. Der Saponit scheint sich durch hydrothermale Umwandlung gebildet zu haben, wahrscheinlich während des Pliozäns. Das Material zeigt in den Röntgenpulverdiffraktogrammen eine 061 Peak bei 1,529 Å. Sein  $Al_2O_3$ - bzw.  $MgO$ -Gehalt beträgt um 4,4% bzw. 23,5%. Die Summe der oktaedrisch koordinierten Atome der halben Elementarzelle beträgt um 2,82. Der Saponit scheint aus einer einzigen, einheitlichen Tonart zu bestehen; die Hauptverunreinigungen sind feine Diopside- und Tremolith-splitter. Die Infrarot- und thermischen Eigenschaften des Ballarat-Saponits sind ähnlich denen des Allt Ribhein-Saponits. Er hat eine niedrigere Wasseraufnahme-Kapazität als Montmorillonit und ist durch niedrigere Atterberggrenzen und Expansionsdrücke sowie durch höhere Kompaktionsdichten charakterisiert. Die offensichtliche Dichte des Saponits von  $2,865 g/cm^3$  ist größer als die von Montmorillonit. Dieser Saponit ist vom Source Clays Repository der Clay Minerals Society erhältlich. [U.W.]

**Résumé**—De la saponite blanche se trouve dans des joints et des zones de fracture ouvertes dans de la roche calcaire dolomitique métamorphosée près de Ballarat, Californie. La saponite semble s'être formée par altération hydrothermale, possiblement à l'époque Pliocène. Le matériau montre un sommet de diffraction 06/ à 1,529 Å, des contenus en  $\text{Al}_2\text{O}_3$  et  $\text{MgO}$  d'à peu près 4,4 et 23,5%, respectivement, et une valeur de somme de demi-maille d'atomes octaédraux d'à peu près 2,82. La saponite semble consister en une seule espèce d'argile uniforme, les impuretés principales sont de fines échardes de diopside et de trémolite. Les propriétés infrarouges et thermiques de la saponite de Ballarat sont semblables à celles de la saponite Allt Ribhein. Elle a une capacité de rétention d'eau plus basse que la montmorillonite, et est caractérisée par des limites Atterberg et des pressions d'expansion plus basses, et des densités de compaction plus élevées. La densité apparente de la saponite, 2,865  $\text{g/cm}^3$ , est plus grande que celle de la montmorillonite. Cette saponite est disponible au Répositoire d'Argiles de Source du Clay Minerals Society. [D.J.]