LOUGHLINITE, A NEW HYDROUS SODIUM MAGNESIUM SILICATE¹

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Abstract

A new hydrous sodium magnesium silicate from the Green River formation in Swertwater County, Wyo., having the formula $Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O$, is named after Dr. Gerald F. Loughlin, former Chief Geologist of the U. S. Geological Survey.

The mineral is found in dolomitic oil shale and in thin section has been seen replacing shortite, northupite, and searlesite.

Loughlinite is a fibrous, pearly-white mineral with a silky luster and is virtually identical to sepiolite (4MgO·6SiO₂·8H₂O), both in hand specimen and microscopically. Loughlinite may be distinguished from sepiolite by the 8 per cent Na₂O in the structure and by comparison of the x-ray fiber diagrams. The indices of refraction of loughlinite are $\alpha = 1.500$, $\beta = 1.505$, and $\gamma = 1.525$.

Prolonged leaching of loughlinite with water or immersion of loughlinite in magnesium salt solutions converts the mineral into a material that is apparently identical to sepiolite.

Introduction

During the logging of the drill core of the John Hay, Jr., Well No. 1, Sweetwater County, Wyoming, in 1940, in which the new minerals shortite (Fahey, 1939) and bradleyite (Fahey and Tunell, 1941) were found, a new asbestos-like mineral, a hydrous sodium magnesium silicate, was found in quantities too small for a thorough study. The same mineral was observed in 1946 in the drill core of the nearby Union Pacific Well No. 4, but here too in sparse amounts. However, in the summer of 1947, many specimens of dolomitic shale containing this fibrous mineral were obtained from material excavated during the sinking of the shaft of the Westvaco Chlorine Products Corporation that was put down for the purpose of mining trona. These three locations are between 18 and 20 miles west of the town of Green River, Sweetwater County, Wyoming. It was not until recently that sufficient material was purified for chemical analysis and related studies.

Loughlinite (Fahey and Axelrod, 1948) was first announced in a paper given at the 28th annual meeting of the Mineralogical Society of America that was held in Ottawa, Canada, in December 1947. The name loughlinite has been given to the new mineral in honor of the late Dr. Gerald F. Loughlin, who, during his many years as Chief Geologist of the U. S. Geological Survey, did much to encourage research in mineralogy.

PHYSICAL PROPERTIES

Loughlinite is a fibrous, pearly-white mineral with a silky luster, and is

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TABLE 1		CHEMICAL.	ANALYSES
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	Loughlinite (le	Loughlinite (leached 58 days)		Sepiolite			
	A	A'	Molec- ular ratios	В	Molec- ular ratios	С	Molec- ular ratios
SiO ₂	50.80	50.80	6.00	51.52	4.97	52.50	6.00
Al_2O_3	0.66	0.66	0.05	0.75	0.04	0.60	0.04
Fe_2O_3	1.85	1.85	0.08	2.64	0.10	2.99	0.13
FeO	1.51	1.51	0.15	1.35	0.11	0.70	0.07
TiO_2	0.02	0.02	0.00	0.02	0.00	=	-
CaO	0.12*-0.12=	0.00	0.00	0.02	0.00	0.47	0.06
MgO	16.18 - 0.17 =	16.01	2.82	19.60	2.82**	21.31	3.63
Na_2O	8.16	8.16	0.93	0.06	0.01	-	-
K_2O	0.00	0.00	0.00	0.00	0.00	-	-
H ₂ O – 110° C _* H ₂ O + 110° C.	13.68 6.82	13.68	8.08	13.38	7.66	21.27	8.10
CO_2	0.30*-0.30=	0.00	0.00	0.00	0.00	_	
				-		-	
	100.10 dolomite	0.38		99.76		99.84	
	magnesite	0.21					
		100.10					

- A Loughlinite (leached 6 hrs.), J. J. Fahey, analyst.
- A' Loughlinite (leached 6 hrs.) after deduction of dolomite and magnesite.
- B Loughlinite (leached 58 days), J. J. Fahey, analyst.
- C Sepiolite from Ampandrandava, Madagascar (Caillère, 1951, p. 230).
- * Calculated as dolomite and magnesite.

virtually identical in appearance to sepiolite, $4 \text{MgO} \cdot 6 \text{SiO}_2 \cdot 8 \text{H}_2 \text{O}$, both in a hand specimen and microscopically. The physical and optical constants were determined, and the chemical analysis was made (Table 1, Sample A) on material that had been leached for 6 hours to remove contaminating sodium carbonate, and air dried at room temperature for 10 days.

The specific gravity of loughlinite is 2.165. This was measured by means of a fused silica Adams-Johnston pycnometer, using distilled water. Owing to the extreme fineness of the fibers no effort was made to determine hardness. The indices of refraction of loughlinite are:

$$\alpha = 1.500$$

^{**} The molecular ratio of MgO is arbitrarily given the value 2.82.

 $\gamma = 1.525$ 2V = 60° positive (calculated)

The indices were measured on randomly oriented bundles of tiny fibers. By making very many measurements a close approximation of the true value of α , β , and γ is believed to have been made. Loughlinite has parallel extinction and positive elongation. A thin section cut normal to the long axis of a bundle of the fibers gave a positive acute interference figure. No twinning was observed.

Loughlinite which had been leached for 58 days (Table 1, analysis B) was so matted and finely divided that only $\alpha = 1.520$ and $\gamma = 1.532$ could be measured.

Differential thermal analyses of the natural and the treated lough-linites have been made by our colleague George T. Faust who will report elsewhere on his studies. His data show that loughlinite gives a DTA curve similar to that of sepiolite. The studies of Caillère and Henin (1947) showed that the DTA curves of sepiolite and palygorskite could be altered by treatment with various salt solutions previous to thermal analysis. The leaching experiments performed in the study in purifying the mineral are equivalent to treatments in salt solution.

PARAGENESIS

Loughlinite is found in medium- to low-grade oil shale of the Wilkins Peak member of the Green River formation. It was formed after compaction of the sediments had taken place, as is indicated by the absence of flow lines in the shale. Loughlinite is found in veins, usually less than 3 cm long, that vary in thickness up to 1 cm, which is the maximum length of the fibers.

In thin section, it is readily seen that loughlinite replaces the dolomitic shale, isolated small islands of the shale being frequently observed within the area occupied by loughlinite. Shortite $(Na_2CO_3 \cdot 2CaCO_3)$, northupite $(Na_2CO_3 \cdot MgCO_3 \cdot NaCl)$, and searlesite $[NaB(SiO_3)_2 \cdot H_2O]$ are likewise seen being replaced by loughlinite. In no instance has loughlinite been observed in the process of replacement by another mineral.

CHEMICAL PROPERTIES

The sample was prepared by carefully handpicking the fibrous mineral from vugs opened up on freshly broken surfaces of the dolomitic shale. It was not necessary to use a binocular microscope in this operation, except in the final examination of the picked material. Care was taken to free the ends of the fibers from small particles of adhering shale. All shale except a quantity estimated at less than one per cent was finally removed

by agitating, with a glass stirring rod, the fluffy fibrous material in a large beaker. After about 20 minutes of this treatment the loughlinite was removed, leaving fine particles of shale in the bottom of the beaker. This operation was repeated several times until no residual shale remained in the beaker.

Wilkins Peak member of the Green River formation, wherein loughlinite is found, contains throughout its vertical column, varying small quantities of sodium carbonate, so finely disseminated as to be undetectable by means of the petrographic microscope, even when using an oil-immersion objective. Addition of a drop of acid confirmed the presence of carbonate in the prepared sample of loughlinite, and a quantitative determination showed 1.72 per cent CO₂ to be present. Electron diffraction powder patterns indicated that a small amount of extraneous material was present in the prepared loughlinite sample. This extraneous material could not be positively identified.

Most of the carbonate was removed by placing the fluffy asbestos-like material (approximately 17 grams) in a one-liter Erlenmeyer flask, adding about 700 ml. of distilled water at room temperature, shaking well and allowing to remain for 6 hours. After filtering with suction on a Buchner funnel the sample was air-dried for about 10 days. The mat that had formed on the funnel was then shredded to its former fluffy condition, allowed to air-dry for another week, and was then ready for analysis (Table 1, analysis A).

Analysis A shows that some carbonate is still present after 6 hours of leaching. It is known that a small amount of shale containing dolomite and magnesite is present in the prepared sample. The residual 0.30 per cent CO_2 was then assumed to be from these contaminants.

The x-ray fiber diagrams of loughlinite taken before and after the 6-hour leaching process are identical.

It was found that prolonged leaching at room temperature gradually removed sodium and probably silica from loughlinite. A sample of approximately 12 grams of the analyzed sample of loughlinite (Table 1, analysis A) was placed in a 1 liter Erlenmeyer flask, 200 ml. of distilled water was added, and after shaking well, it was allowed to remain (stoppered) for 5 days at room temperature. The water was then filtered off, and the sample air-dried. Upon analysis the per cent Na₂O was found to have dropped from 8.16 to 6.96. The sample was then returned to the flask and 200 ml. of water added. The flask was stoppered, well shaken, and allowed to remain for an additional 8 days, or a total of 13 days of leaching. The analysis of the sample after filtering and air-drying showed that the Na₂O content had been reduced to 5.60 per cent. This leaching process was repeated for an additional 45 days, making a total leaching

time of 58 days. The leached and dried sample then contained only 0.06 per cent Na_2O (Table 1, analysis B), and gave an x-ray fiber diagram identical to that of sepiolite.

A comparison of analysis A' with analysis B shows that the Mg/Si ratio increased upon leaching for 58 days. It is apparent then that either Si was removed or that Mg was added to the loughlinite structure during this leaching process. It is improbable that magnesium was added to the structure since only distilled water (approximately 2000 ml.) was used in the leaching process. It was also noted that colloidal silica was present in some of the wash water after leaching.

A sample of loughlinite was treated for 7 days in a small amount of 1 per cent $\mathrm{MgCl_2}$ solution and was then washed with a small amount of distilled water. The resulting material gave an x-ray fiber diagram identical to that of sepiolite. This experiment, later repeated with 200 ml. of 2 per cent $\mathrm{Mg(NO_3)_2}$ solution, gives the same results.

Samples of loughlinite which had been leached for 58 days or which had been treated with a 1 per cent MgCl₂ solution for 7 days, were treated with cold 30 per cent NaCl solution for 11 weeks. Similar samples were treated for 9 days with a cold saturated solution of Na₂CO₃. Neither treatment resulted in a reversion to the original loughlinite as shown by the x-ray fiber diagrams.

The 13.68 per cent of water lost from loughlinite at 110° C. (Table 1, analysis A) was completely regained after 8 hours at room temperature and humidity.

Under similar conditions sepiolite regains about 80 per cent of the water lost at 110° C.

Loughlinite is differentially soluble in hydrochloric and sulfuric acids, leaving a siliceous skeleton that retains the shape of the fibers. When heated to 900° C. incipient fusion takes place.

X-RAY STUDIES

Three types of x-ray patterns were made in this study. These are:

- 1. X-ray fiber diagrams taken in a rotation camera by rotating a small bundle of fibers about the fiber axis (c). These patterns show the hk0, hk1, hk2, hk $\overline{1}$ and hk $\overline{2}$ nets of loughlinite and sepiolite.
- 2. X-ray zero-level fiber diagrams taken in a powder camera with a radius of 57.3 mm. These patterns show only the hk0 net of loughlinite and sepiolite.
- 3. X-ray powder patterns. In an attempt to minimize the preferred orientation the x-ray powder spindle was made from fibers that had been rolled into a ball mount (Hildebrand, 1953).

X-ray fiber diagrams taken in a rotation camera show the following:

- 1. Untreated loughlinite is identical to loughlinite (leached 6 hours).
- 2. Loughlinite (leached 6 hours) definitely has a different unit-cell than that of sepiolite from Ampandrandava, Madagascar.
 - 3. Loughlinite (leached 58 days) is identical to sepiolite.
- 4. Loughlinite (leached 6 hours then treated 7 days in MgCl₂ solution) is identical to sepiolite.

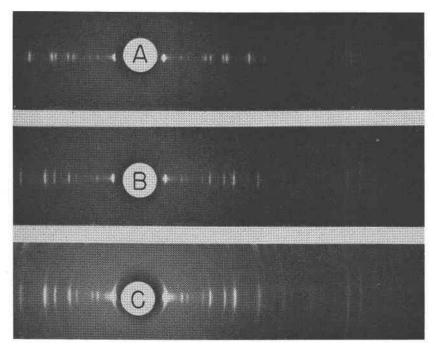


Fig. 1. Zero-level fiber diagrams of

- A) loughlinite, Sweetwater Co., Wyo. (leached 6 hrs.), Table 1, analysis A.
- B) sepiolite, Ampandrandava, Madagascar, Table 1, analysis C.
- C) loughlinite, Sweetwater Co., Wyo. (leached 58 days), Table 1, analysis B.
- 5. The dimension along the fiber direction (c) is 5.3 ± 0.1 Å for both loughlinite and sepiolite.

To further illustrate the similarities and differences of loughlinite (leached 6 hours), loughlinite (leached 58 days), and sepiolite, and also to obtained more accurate values for the spacing of the hk0 reflections, zero-level fiber diagrams were made in an x-ray powder camera using copper radiation. The patterns of these materials can be seen in Fig. 1. The spacings of the hk0 reflections measured from these three patterns are tabulated in Table 2. These measurements definitely show that

Table 2. X-Ray Data Measured from the Zero-Level (l=0) Fiber diagrams† of loughlinite and sepiolite $CuK\alpha$ (Ni filter), $\lambda = 1.5418$ Å, cut off at $d_{\rm obs.} = 13$ Å.

Loughlinite Sweetwater County, Wyo. (Leached 6 hours) (Table 1, analysis A)		Mad	pandrandava, agascar analysis C)	Loughlinite Sweetwater County, Wyo (Leached 58 days) (Table 1, analysis B)		
$d(ext{Å})$	1*	$d(ext{Å})$	I*	d(Å)	I*	
12.8	≫100	12.0	≫100	12.0	≫100	
10.2	4	9.6	9	9.6	9	
7.6	35	7.5	50	7.5	50	
6.4	13	6.7	13	6.7	25	
==	_		-	6.0	2	
5.01	18	5.02	25	5.02	25	
4.80	50		-	_	-	
4.45	100	4.52	100	4.52	100	
4.28	6	4.44	4	4.40	6	
3.79	100	3.75	70	3.75	70	
3.65	100	3.46	6	3.45	6	
3.59	35	3.37	70	3.34	100	
3.08	13	0.01	_	_		
2.90	70	2.94	4		8-	
2.82	6	2,71		-		
2.77	4	2.69	35	2,68	35	
2.56	13	2.59	2	2.57	5	
2.44	2	2.42	3	2.40	5	
2.44	2	2.25	13	2.24	9	
2.13	4	2.14	6	2.14	6	
2.13	1	2.14	0	2.06	2	
1.90	13	1.92	4	1.91	6	
1.90	13	1.88	3	1.87	6	
	 -	1.82	3	1.81	6	
	_	1.73	2	1.01	_	
1 ((0	_		2	1.694	2	
1.668	9	1.690 1.642	2	1.641	2	
1.644	2	1.042	Z	1.041	2	
1.623	4	1.594	2		20000	
		1.594	18	1.583	25	
1 550	10	1.381	10	1.363	2.5	
1.559	18	4 502	12	1.506	18	
4 404	_	1.503	13	1.300	10	
1.484	6			-		
1.463	4		-	1.415	2	
1.414	3	-		1.415	4	
1.383	3	4 272	-		6	
		1.372	3	1.370	6	
		1.352	4	1.344	0	

^{*} Intensities were estimated visually with a calibrated intensity strip.

[†] Fiber diagrams were taken in an x-ray powder camera, radius = 57.3 mm.

loughlinite (leached 6 hours) has a different unit-cell than that of sepiolite whereas loughlinite (leached 58 days) has a unit-cell identical to that of sepiolite.

A zero-level fiber diagram was also made of loughlinite (leached 6 hours) which had been immersed in 200 ml. of 2 per cent Mg(NO₃)₂ solution for 10 days. This pattern is identical to the zero-level fiber diagram of sepiolite shown in Fig. 1B.

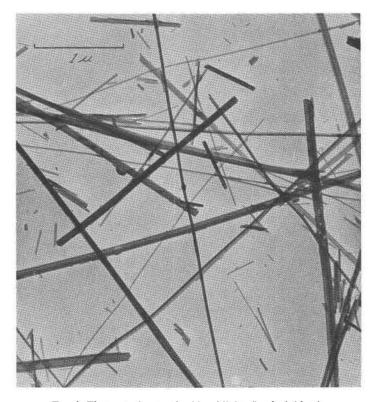


Fig. 2. Electron micrograph of loughlinite (leached 6 hrs.).

The x-ray powder data of loughlinite (leached 6 hours), loughlinite (leached 58 days), and sepiolite are compared in Table 3. These powder patterns are somewhat similar in appearance and are not as diagnostic of phase differences as are the fiber diagrams. The differences in the powder patterns of loughlinite (leached 6 hours) and for the sepiolite from Ampandrandava indicate that they are not the same mineral. The powder pattern of loughlinite (leached 58 days) and that of sepiolite from Ampandrandava show no greater differences than are found in

Table 3. Measured X-Ray Powder Data for Loughlinite and Sepiol CuK α (Ni filter, $\lambda\!=\!1.5418$ Å; cut off at $d_{\rm obs.}\!=\!13$ Å

Loughlinite Sweetwater County, Wyo. (Leached 6 hrs.) (Table 1, analysis A)		Sepic Ampandrandav (Table 1, a	a, Madagascar	Loughlinite Sweetwater County, Wyo (Leached 58 days) (Table 1, analysis B)		
d (Å)	I*	d (Å)	I*	d (Å)	I*	
12.9	100	12.3	100	12.3	100	
7.63	5	7.56	8	7.56	3	
6.46	1b	6.73	4	6.73	1.5b	
-	t-	-	-	6.11	1	
5.04	1.5	5.05	6	5.02	3b	
4.81	5	4.81	<1	4.78	1.5	
4.51	7b	4.54	14	4.53	6	
4.34	18n	4.32	13	4.32	18n	
4.15	1.5	-	====	4.17	2	
4.00	1		-	3.99	1.5	
3.83	6b	3.77	14	3.77	9	
3.63	7b	3.55	3	3.55	3	
3.31	5	3.36	15	3.36	6	
3.21	1	3.20	8	3.20	5b	
3.09	2 5	3.06	3	3.05	1.5b	
2.92	5	2.84	2b	2.84	2n	
-	-	2.70	6b	2.69	2b	
2.64	6 5	2.63	6	2.63	3	
2.60	5			2.59	6n	
2.55	7n	2.57	18	2.56	9n	
2.48)	5)	2.45	6b	2.45	6	
2.44	5)	2.41	6b	2.39	2	
2.32)	2)	2.27	8b	2.27	5b	
2.23	15		-	2.21	1	
2.16)	3)	0.12	2.7	0.42	4 5	
2.11	3∫	2.13	36	2.13	1.5	
2.06)	1)	2.07	6b	2.07	2	
2.02	15	1.971	3)	1.996	1	
1.907	1.5	1.881	35	1.881	$\frac{1}{b}$	
1.804) 1.774	1.5	1.821	1	1.817	1b	
1.732	$\frac{1.5}{2}$	1.766	1	1.728	1	
1.695	3)	1.704	$\overline{3b}$	1.697	2	
1.675	3	1.672	<1	1.669	1	
1.634)	1	1.637	<1b	1.629	$\overset{1}{1}b$	
1.608	1	1.598)	6)	1.598)	2)	
1.567	2	1.584	6	1.578	2	
1.550	3	1.554	$\stackrel{(0)}{2b}$	1.549	3	
1.517	2 3 3	1.522	$\frac{20}{2n}$	1.519)	4n \	
1.491	1	1.507	$\frac{2n}{3}$	1.503	1.5	
1.471	1	1.471	<1	1.467	1.5b	
1.413	î	1.414	3b	1.414	1.5b	
1.380	$\frac{1}{1b}$	1.374	16	TIT,	= 1.50	
1.318)	1)	1.354	1	1.349	1b	
1.293	$\{1.5\}$	1.339	1	1,312	2	
2,270)	1.0)	1.301	$\frac{1}{4b}$	1.298	3	
		1,287	1	1.262	1.5	
		1.005	$\frac{1}{1}b$. 202	1.0	

^{*} Intensities were estimated visually with a calibrated intensity strip. b = broad line.

n = narrow line.

 $^{\} =}$ band, d values for measurements at each edge of band.

patterns of four fibrous sepiolites from Ampandrandava (U.S.N.M. 106827), Tsing Shi-Ling, Inner Mongolia (U.S.N.M. 90719), Chester Co., Pa. (U.S.N.M. 85890), and Little Cottonwood Canyon, Utah (U.S.N.M. C-3808).

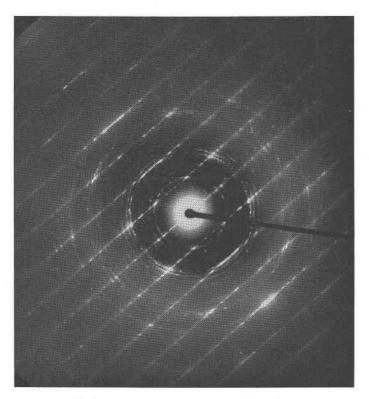


Fig. 3. Electron diffraction fiber diagram of sepiolite from Ampandrandava, Madagascar.

ELECTRON MICROSCOPE AND ELECTRON DIFFRACTION STUDIES

Electron micrographs of untreated loughlinite, loughlinite (leached 6 hrs., Fig. 2), loughlinite (leached 58 days), and sepiolite from Ampandrandava, Madagascar, show that the habits are identical. The width of the individual fibers of loughlinite appear to be about 70 Å.

Electron diffraction powder patterns of loughlinite (leached 6 hrs.) and of sepiolite show small differences similar to those found in the x-ray powder patterns. Electron diffraction fiber diagrams, obtained by orienting a very small bundle of fibers with the fiber direction approximately normal to the electron beam, show that the dimension along the

fiber direction (c) is 5.26 ± 0.01 Å. in both minerals. Fig. 3 shows such a fiber diagram obtained from a bundle of sepiolite fibers. The rings in this figure are given by those fibers which are randomly oriented.

DISCUSSION

The molecular ratios of loughlinite (leached 6 hrs., analysis A', Table 1) indicate an ideal formula of $Na_2O\cdot 3MgO\cdot 6SiO_2\cdot 8H_2O$. The ideal formula for sepiolite, as indicated by the molecular ratios given in Table 1, and as suggested by Caillére (1933), Schaller (1936), and Kauffman (1943), is $4MgO\cdot 6SiO_2\cdot 8H_2O$. The molecular ratios for the material remaining after leaching loughlinite for 58 days (analysis B, Table 1) appears to be explained by assuming that silica is slowly leached, along with sodium. The resultant leached product has the apparent formula of $3MgO\cdot 5SiO_2\cdot 8H_2O$.

The experimental work done indicates that loughlinite can be altered to sepiolite (or to a substance closely resembling sepiolite) by two different routes. A comparison of analyses A' and B (Table 1) indicates that sodium and silica is removed from the loughlinite structure by prolonged leaching, *i.e.*,

$$\begin{array}{c} Na_2O \cdot 3MgO \cdot 6SiO_2 \cdot 8H_2O \xrightarrow{prolonged} 3MgO \cdot 5SiO_2 \cdot 8H_2O + Na_2SiO_3 \\ & \text{(loughlinite)} \end{array}$$
 (sepiolite-like substance)

The alteration of loughlinite to sepiolite or to a sepiolite-like substance on immersion in magnesium salt solutions suggests a simple substitution of 2Na for Mg, *i.e.*,

$$\begin{aligned} \text{Na}_2\text{O}\cdot 3\text{MgO}\cdot 6\text{SiO}_2\cdot 8\text{H}_2\text{O} &\xrightarrow{\text{Mg}^{++}} 4\text{MgO}\cdot 6\text{SiO}_2\cdot 8\text{H}_2\text{O} \ + \ 2\text{Na}^+ \end{aligned}$$
 (loughlinite) (sepiolite-like substance)

It is difficult to imagine a crystal structure which might accommodate transformations of the above nature; thus until further information is obtained the relationship of sepiolite and loughlinite will remain in question.

ACKNOWLEDGMENTS

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