

A RESTUDY OF STEVENSITE AND ALLIED MINERALS*

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ABSTRACT

Stevensite was shown by Faust and Murata to be a montmorillonite group mineral. Brindley subsequently proposed that stevensite consists of an interlayered talc-saponite mineral. A restudy of stevensite shows that it is not an interlayered talc-saponite mineral but rather a type of defect structure. The defect structure arises from a deficiency in the total number of ions in octahedral coordination. This concept of stevensite as a defect structure seems to fit all of the data from *x*-ray, infrared, differential thermal analysis, and solution studies.

Ghassoulite is shown to contain 0.36% Li₂O and 3.22% F. Experimental studies show it to be like hectorite instead of stevensite.

Synthetic stevensites, the interstratified "saponite-talc" of Alietti, and hanušite are interpreted in terms of the results reported here.

Two new chemical analyses are reported:—Ghassoulite from Djebel Ghassoul, Morocco, and a mixture containing stevensite from Mine Creek, North Carolina.

It is recommended that the names hanušite (=stevensite+pectolite) and ghassoulite (=hectorite) be relegated to the synonymy.

INTRODUCTION

Stevensite was redefined as a member of the montmorillonite group by Faust and Murata (1953) on the basis of differential thermal analysis, staining, *x*-ray powder diffraction, and chemical studies. Since that time further studies have appeared on stevensite and related minerals and the original literature on hanušite has become available to us. This paper presents the results of further studies and an interpretation of the various data.

During World War II, Kašpar (1942) described the mineral hanušite which he characterized as the magnesium end-member of the supposed pectolite-walkerite series. Fleischer (1943), commenting on the new mineral name, hanušite, suggested the relationship to stevensite. Brindley (1955) made further studies on stevensite and proposed on the basis of his *x*-ray data that stevensite "may be described as a talc-saponite inter-layered mineral."

Millot (1954), unaware of the paper by Faust and Murata, gave the name ghassoulite to a clay from Morocco which is used in laundering. This clay had been described, with an analysis, by Damour (1843). Fleischer (1955) in his discussion of new mineral names, stated that ghassoulite was probably identical with stevensite. Andrea Alietti (1956) described a mineral related to stevensite from Monte Chiaro, Italy, as an interlayered mineral of saponite and talc.

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DESCRIPTION OF THE SPECIMENS STUDIED

The mineral cabinet of Albert R. Leeds, (the discoverer of stevensite) was bequeathed to the Stevens Institute of Technology at Hoboken, New Jersey, where Leeds had been a professor. In an attempt to locate the type specimen of stevensite, we requested Professor S. C. Williams of that institution to search for it in the Leeds cabinet. Unfortunately, neither the type material nor any specimen of stevensite could be located. Professor Williams did, however, send us a specimen of stevensite which he had obtained for this study from William C. Casperson, at that time curator of minerals in the Paterson Museum, Paterson, New Jersey.

Color references are to Ridgway's standards symbols.

Stevensite No. 1.—Locality: Springfield, New Jersey (U.S.N.M. R4719). Pseudomorphous after pectolite. Pink masses. The powder of the sample used for the chemical analysis and other studies is pale Salmon Color, 9'.OR·O.f. Analysis 1, Table 1.

Geological Occurrence: Late stage hydrothermal replacement of pectolite in cavities in basalt.

Stevensite No. 2.—Locality: Jersey City, New Jersey, (Selfridge No. 51). Deweylite, "Serpentine after pectolite." Color, Wood Brown, 17''' O. Y.

Geological Occurrence: Late stage hydrothermal replacement of pectolite in cavities in basalt. From Professor Paul F. Kerr.

Stevensite No. 3.—Locality: Paterson, New Jersey (F-1104). Pseudomorphous after pectolite. Color of fresh surface is Pale Vinaceous-Fawn 13''' O. Y.-O.f. Color of weathered surface is Vinaceous-Buff, 17''' O. Y.d.

Geological Occurrence: Late stage hydrothermal replacement of pectolite in cavities in basalt. Collected by George T. Faust.

Stevensite No. 4.—Locality: New Street Quarry, Paterson, New Jersey (#685). Pseudomorphous after pectolite. Color of most of the specimen is pale pinkish buff, 17'' O. Y.f. A small portion is safrano pink, 7. R. O. f.

Geological Occurrence: Late stage hydrothermal replacement of pectolite in cavities in basalt. From Professor S. C. Williams.

Stevensite No. 5.—Locality: Mine Creek, Bakersville, Mitchell County, North Carolina (F-48). Insufficient material for naming the color. This sample is impure and contains some serpentine mineral.

Geological Occurrence: Probably a hydrothermal alteration product in dunite. From Dr. C. S. Ross. Analysis 3, Table 1.

Stevensite.—Synthetic. Prepared by N. L. Bowen and O. F. Tuttle (1949). Tentatively identified by them as sepiolite (?). Charge T-2-37. Theoretical Composition of sepiolite-held at 340° C. and at a pressure 30,000 pounds per square inch. Color white.

Ghassoulite.—Locality: Djebel Ghassouel* mine, on the spur of the Moyen Atlas Mountains which dominate the valley of the Moulouya, Ksabi Province, Eastern Morocco. Color—light drab, 17''' O. Y.b. Analysis 4, Table 1.

Geological Occurrence: Tertiary sedimentary argillite. Continental facies. Series include periodically: flint, the "earth ghassoulite," green marl and white limy-marl. Ghassoulite is authigenic in origin in a lake deposit of chemical sediments (Jeannette, 1952; Millot, 1949).

Hectorite.—Locality: About 3 miles south of the village of Hector, San Bernardino County,

* Some transliterations give Rhassoul and Rassoul.

TABLE 1. CHEMICAL ANALYSES OF STEVENSITE, GHASSOULITE, AND HECTORITE

Analysis No.	1	2	3	4	5	6	7	8
Name	Stevensite No. 1	Stevensite No. 2	Stevensite No. 5	Ghassoulite	Ghassoulite	Hectorite	Hanušite	"Talc- saponite"
Locality	Springfield, N. J. U.S.N.M. R4719	Jersey City, N. J. Selfridge No. 51	Mine Creek, Bakersville, N. C. F-48	Djebel Ghas- souel mine Ksabi; Prov- ince, Morocco	Djebel Ghas- souel mine Ksabi; Prov- ince, Morocco	Near Hector, San Bernardino County, Calif.	Liebstadt, Riesengebirge	Monte, Chi- aro, Val di Taro, Italy
SiO ₂	57.30	57.24	47.0	55.02	55.0	55.86	57.37	54.31
Al ₂ O ₃	None	None	—	1.12	1.2	.13	—	1.90
Fe ₂ O ₃	0.32	1.14	—	—	1.4	.03	—	—
FeO	None	0.12	5.5	0.70	—	—	3.57	2.95
MnO	0.21	0.75	—	—	—	—	0.54	n.d.
MgO	27.47	27.89	29.7	24.89	28.0	25.03	18.55	27.44
CaO	0.97	0.38	0.2	0.54	1.01	Tr	4.84	1.01
K ₂ O	0.03	—	—	0.43	0.52	.10	—	0.86
Na ₂ O	0.03	—	—	0.94	—	2.68	—	0.32
Li ₂ O	—	—	—	0.36	—	1.05	—	—
CO ₂	—	—	—	0.30	—	—	—	—
F	None	None	—	3.22	—	—	—	—
H ₂ O ⁺	7.17	7.69	—	6.42	—	5.96	6.49	9.48
H ₂ O ⁻	6.69	4.76	17.7	7.66	10.35	2.24	8.68	2.59
Σ	100.19	99.97	100.1	101.68 ¹	97.48 ²	102.98 ³	100.04	100.86
Analyst	K. J. Murata	K. J. Murata	C. Milton	J. J. Fahey	A. A. Damour	R. E. Stevens	J. V. Kašpar	A. Alietti

¹ 101.68-1.36 (0=F) = 100.32. Contains 0.08 TiO₂.² Excludes 1.5% sand reported in the original.³ 102.98-2.51 (0=F) = 100.47.

California. Color pure white. API clay mineral standard, H-34, used in *x*-ray studies.

Analysis and DTA study on other purified samples.

Geological Occurrence: Hydrothermal replacement of tuff enclosed within basaltic lava flows. Analysis 6, Table 1.

Table 1 contains in addition to the analyses cited above the analysis of other minerals pertinent to this discussion. These are analysis 2, stevensite from Jersey City, N. J. (Faust and Murata, 1953); analysis 5, ghassoulite from Morocco (Damour, 1843); analysis 7, hanušite from the Riesengebirge (Kašpar, 1942) and analysis 8, talc-saponite from Italy (Alietti, 1956). Analysis 3, stevensite from North Carolina and analysis 4, ghassoulite from Morocco are new.

DIFFERENTIAL THERMAL ANALYSIS DATA

The differential thermal analyses were made in a modified form of the Alexander, Hendricks, and Nelson (1939) apparatus. The samples were heated in a nickel block at the rate of 12° C. per minute. The records were obtained as photographs. The samples were all air-dried at room temperature and at a relative humidity of about 65%.

DTA patterns of the original sample, stevensite No. 1, studied by Faust and Murata (1953); of stevensite No. 4, from Paterson, New Jersey; of stevensite No. 5, an impure stevensite from Mine Creek, North Carolina; of synthetic stevensite synthesized by Bowen and Tuttle (1949); of ghassoulite from Morocco; and of hectorite from Hector, California are shown in Fig. 1. The general similarity of these patterns is apparent and they are typical of the montmorillonite group of minerals. The sample weights and data obtained from the curves are summarized in Table 2.

The DTA record (C-123) of stevensite No. 1 covers the temperature range of 25° C. (at the beginning of the curve) to 1061° C. (at the end of the curve). At the high temperature end of the curve from about 885° C. to 1061° C. the record is essentially a straight line. It is within this region that talc undergoes its dehydration and its endothermic trough generally has its minimum at about 950° C. to 1000° C. Such a trough does not appear on the curve for stevensite No. 1. The behavior of stevensite No. 4 (C-785), bearing in mind the difference in weight of the samples, is almost the same as that of stevensite No. 1, and the temperatures of the minima of the troughs are very close (see Table 2). The small area under the low-temperature endotherm of the synthetic stevensite (C-519) arises from the drying out of the sample. Drying out has probably been accelerated by the very fine grain size of the synthetic preparation. The *x*-ray powder pattern of this sample shows two weak lines which suggest the presence of a small amount of chrysotile. Stevensite No. 5, (C-546),

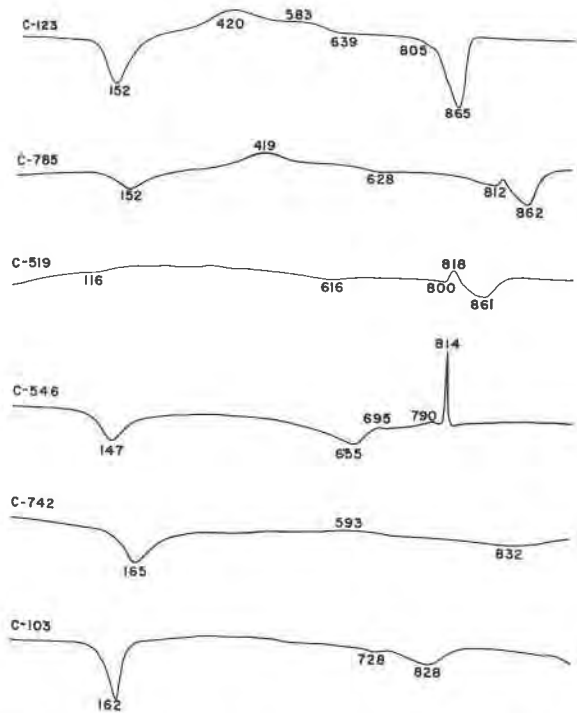


FIG. 1.—Differential thermal analysis curves for Stevensite No. 1, Springfield, New Jersey, C-123; Stevensite No. 4, Paterson, New Jersey, C-785; Synthetic stevensite, Bowen and Tuttle preparation T-2-37, C-519; Stevensite No. 5 with serpentine, Mine Creek, North Carolina, C-546; Ghassoulite, Morocco, C-742; and Hectorite, Hector, California, C-103.

The temperature on the left of the diagram is about 25° C; the temperature at the termination of the curve, on the right, is about 1000° C.

is an impure sample containing a serpentine mineral. The curves of ghassoulite (C-742) and hectorite (C-103) seem most closely alike. It is noteworthy that at the termination of the DTA curve for hectorite a high temperature endothermic trough appears. The onset of this trough is at 1053° C. and as far as this record goes to 1077° C. the minimum was not reached. Such a high temperature endothermic trough in hectorite has been noticed by other investigators, Grim and Rowland (1942) and Schmidt and Heystek (1953).

X-RAY STUDIES

The samples were prepared for x-ray analysis as follows: hectorite was treated with glacial acetic acid diluted with 3 parts of water to remove calcite in the sample, then washed with distilled water. The $<2\mu$ fraction

TABLE 2. OBSERVATIONS ON THE THERMAL ANALYSIS CURVES¹ OF STEVENSITE AND RELATED MINERALS

Mineral	Record no.	Temperature of Troughs and Peaks ° C.						Locality	Weight of sample used (grams)		
		Low temperature		Intermediate temperature		High temperature					
		Endo-thermic	Exo-thermic	Endo-thermic	Exo-thermic	Endo-thermic	Exo-thermic				
Stevensite No. 1	C-123	152	420	639 Inflection	583	805	865	—	—	Springfield, New Jersey U.S.N.M. R4719	0.4762
Stevensite No. 4	C-785	152	419	628 Inflection	—	812	862	—	—	Paterson, New Jersey	0.4513
Stevensite Synthetic with a little chrysoile	C-519	116	—	616	—	800	861	—	—	Bowen and Tuttle Prep. No. T-2-37	0.1862
Stevensite No. 5 with serpentine	C-546	147	—	655	695	—	—	—	790	Mine Creek, North Carolina	0.3022
Ghassoulite	C-742	165	—	—	593	—	832	—	—	Morocco	0.3771
Hectorite	C-103	162	—	—	—	728	828	—	—	Hector, California	0.4377

¹ Resistance in the galvanometer circuit for these curves is as follows: 600 ohms for C-123, C-785, C-742, and C-103; 900 ohms for C-519 and 999.9 ohms for C-546.

was obtained by repeated centrifuging and decanting. Stevensite No. 4 was also fractionated by centrifuging but the Stevensite No. 1 and ghassoulite samples proved too small to handle in this manner. The ghassoulite probably contains a very small amount of quartz. These (Stevensite No. 1 and ghassoulite) were dispersed in distilled water in test tubes by a motor driven brush and allowed to stand until most of the coarser particles had settled. Oriented aggregates of the fine fraction of these four minerals were prepared by pipetting some of the clay suspension onto glass slides or by centrifuging the material onto porous plates by the method of Kinter and Diamond (1956). Randomly oriented powders were prepared by drying the clay at room temperature, crushing to a moderately fine powder and sieving the material with a 200 mesh sieve into aluminum mounts with openings 10×41 mm. Care was taken not to pack the powder tightly in order to avoid introducing too much preferred orientation. X-ray patterns were prepared using a Norelco diffractometer. The operating constants are given below:

	<i>For randomly oriented powders</i>	<i>For oriented aggregates</i>
Radiation	Cu, Ni filtered	Cu, Ni filtered
KVP	40	40
MA	20	20
Divergence slit	4°	1°
Receiving slit	.006 inch	.006 inch
Scatter slit	4°	1°
Sample length	41 mm.	44 mm.
Scale factor	8	16-64
Multiplier	1	1
Time constant	4 sec.	4-2 sec.
Scanning speed	2 °/min.	2 °/min.+1 °/min.
Chart speed	½ inch/min.	½ inch/min.

Traces of the patterns obtained from the randomly oriented powders are shown in Fig. 2. There is a close similarity of the patterns, except for the 00 l diffraction maxima and those due to impurities. The characteristically broad asymmetrical hk bands of the montmorillonite group minerals are present in all four samples.

The differences in these minerals are brought out more clearly in the patterns for the untreated oriented aggregates shown in Fig. 3.

The summary of the diffraction data shown by these two groups of patterns is given in Table 3.

The intensities shown in Table 3 are derived from measurements of the relative areas under the diffraction peaks. Relative intensities for 00 l reflections were based on patterns of oriented aggregates whereas the intensities for the hk reflection were based on the patterns of randomly

oriented powders. The strongest reflection in each of these two groups was assigned an intensity of 10. The reasons for assigning these intensities separately are as follows:

- (1) The $00l$ reflections may vary considerably in intensity with respect to the hk reflections as a result of preferred orientation.
- (2) The wide (4°) slits used with the randomly oriented powder to obtain adequate intensity of the diffracted radiation prevent the recording of diffraction effects below about $5^\circ 2\theta$, and the long spacings of air-dried stevensite occur in this region.

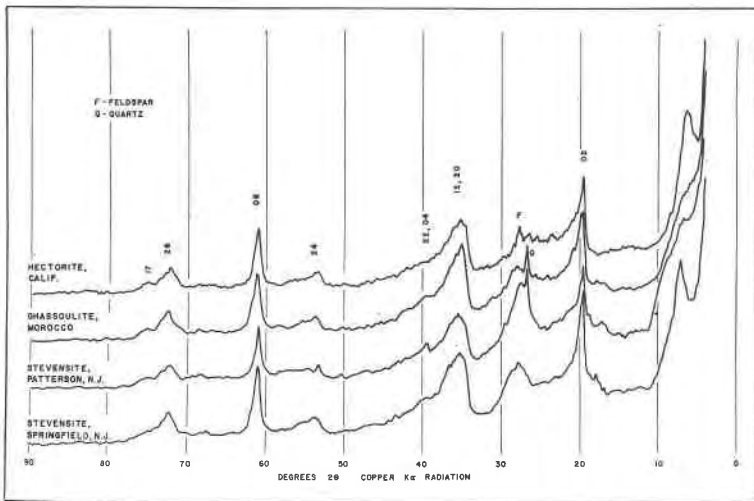


TABLE 3. X-RAY POWDER DIFFRACTION DATA FOR HECTORITE, GHASSOULITE, AND STEVENSITE DRIED AT ROOM TEMPERATURE

Mineral	Hectorite		Ghassoulite		Stevensite No. 4		Stevensite No. 1	
	d (Å)	I* Notes	d (Å)	I* Notes	d (Å)	I* Notes	d (Å)	I* Notes
Locality	Hector, Calif.		Morocco		Paterson, N. J.		Springfield, N. J.	
Indices								
~001	12.7 -14.0	10* variable	12.5 -14.5	10* variable	24.5	1* variable	26.5	2* variable
~002	4.5	6	4.85 - 5.1	<1 variable	12.1 -13.0	10* variable	12.5 -13.0	10* variable
02, 11	3.16 - 3.35	2* variable	4.49	6	4.78 - 5.2	<1* variable	4.94 - 5.0	<1* variable
~003	2.56	10 very broad	3.18 - 3.24	2* variable	4.54	6	4.54	6
13, 20	2.27	1	2.56	10 very broad	3.20 - 3.24	3* variable	3.17 - 3.20	3* variable
04, 22	1.715	2	2.28	1	2.54	10 very broad	2.55	10 very broad
15, 24, 31	1.520	4 sharp	1.710	1	2.28	1	2.28	1
06, 33	1.306	3	1.516	4 sharp	1.720	1	1.710	1
26, 40	1.262	1	1.303	3	1.520	4 sharp	1.520	4 sharp
17, 35, 42			1.262	1	1.307	2	1.306	2
					1.265	1	1.265	<1

* Intensities of 001 reflections based on oriented aggregates.

Intensities of hk reflections based on randomly oriented powders.
Strongest 001 and hk reflections each assigned an intensity of 10.

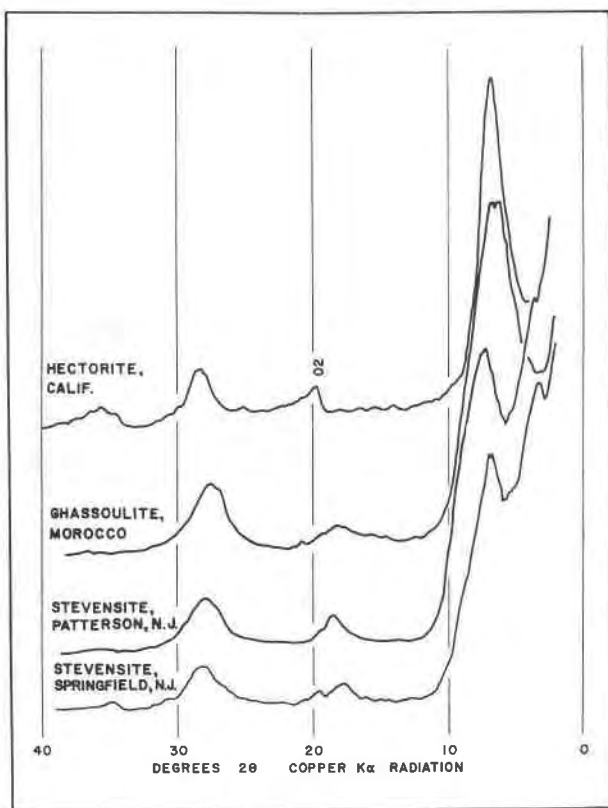


FIG. 3.—X-ray diffractometer patterns of untreated oriented aggregates of hectorite, ghassoulite and stevensite.

No. 4 and Stevensite No. 1. These spacings, together with those in the ranges 12 \AA – 14 \AA , 4.7 \AA – 5.2 \AA , and 3.16 \AA – 3.4 \AA , form a non-integral series, therefore, as Brindley has pointed out, a precise index cannot be assigned to any of these spacings. The $00l$ spacings of ghassoulite and hectorite fall into this category also, as do many montmorillonites in an air-dried state.

Treatment of a montmorillonite with ethylene glycol usually causes expansion of the mineral in the c axis direction to give a basal spacing of $\sim 17 \text{ \AA}$. An x-ray diffraction pattern of such glycol treated material shows this spacing plus an integral series of higher orders from it. Figure 4 shows the patterns obtained from ethylene glycol treated oriented aggregates of hectorite, ghassoulite, and the two stevensite samples. Table 4 lists the spacings for first 6 orders of the $00l$ series. No lines were visible beyond 006.

All samples depart to a greater or lesser degree from a strictly integral series. Brindley (1955, p. 245) has proposed that such departure in stevensite, together with the diffuse nature of the $00l$ reflections, point toward the presence of nonexpanding material interstratified with montmorillonite type layers. He suggests that an interstratification of 9.5–10 Å and 14–15 Å structures could produce the irrational sequence of basal reflections and the 24–25 Å reflection observed in air dried specimens, and

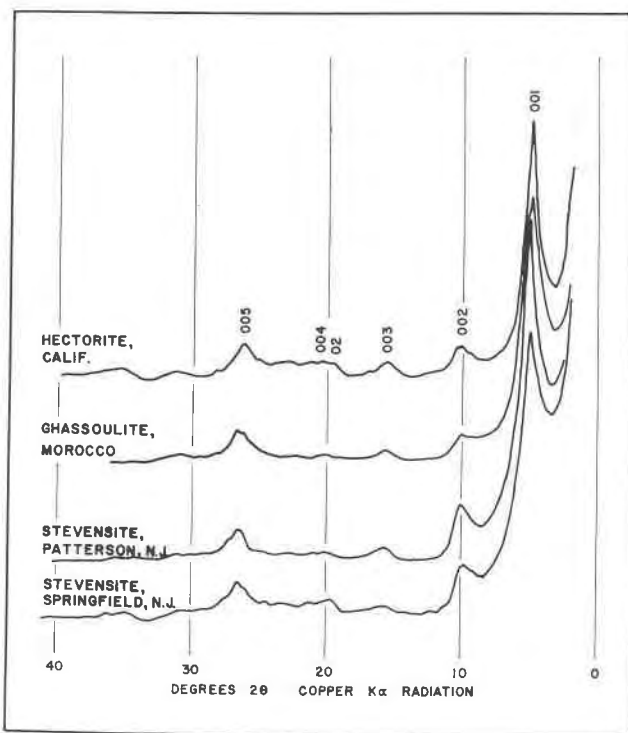


FIG. 4.—X-ray diffractometer patterns of oriented aggregates of hectorite, ghassoulite and stevensite treated with ethylene glycol.

that upon ethylene glycol treatment such a sequence of layers would give “a ‘first’ order at about 17 Å or a little higher spacing, a ‘second’ order at about 9 Å together with higher orders which cannot be reliably identified in the spectrometer traces.” If such is the case for stevensite then both hectorite and ghassoulite might be suspected of containing interstratifications of nonexpanding material. Neither shows a strictly integral sequence of basal orders, nor does either show a less diffuse or better developed sequence.

According to the diagram given by Weaver (1956 p. 206) an assem-

TABLE 4.—X-RAY DATA FOR ORIENTED AGGREGATES TREATED WITH ETHYLENE GLYCOL (Cu/Ni; $\lambda=1.5418 \text{ \AA}$)

Mineral	1. Hectorite	2. Ghassoulite	3. Stevensite No. 4	4. Stevensite No. 1	Calculated
Locality	Hector, Calif.	Morocco	Paterson, N. J.	Springfield, N. J.	$d \text{ (\AA)}$
Indices	$d \text{ (\AA)}$	$d \text{ (\AA)}$	$d \text{ (\AA)}$	$d \text{ (\AA)}$	
001	17.2	18.0	16.9	17.0*	17.0
002	8.8	8.8	8.8	9.0	8.5
003	5.69	5.68	5.69	5.68	5.66
004	?	?	?	?	4.25
005	3.40	3.37	3.39	3.37	3.40
006	2.86	2.90	2.87?	?	2.83

* The 15 Å spacing for ethylene glycol saturated stevensite reported by Faust and Murata (1953, p. 976) was obtained only when the material was also saturated with "duco" cement thinned with acetone.

blage of mixed layered 10 Å and 17 Å units could contain up to about 30% expanded (17 Å) layers without decreasing the first order spacing by more than 0.5 Å. This would suggest that if any nonexpanding units are present in the materials under study, they probably account for less than 30% of any assemblage.

ELECTRON MICROSCOPE STUDIES

Electron micrographs of ghassoulite made at the University of Strasbourg show it appearing as laths and bands analogous to the laths and bands of hectorite. Electron micrographs of stevensite, on the other hand, resemble in morphology the common montmorillonites being irregular shaped thin plates and curled thin fragments (Brindley, 1955).

INFRARED ABSORPTION ANALYSIS

In order to test Brindley's (1955, p. 246) proposal that talc layers constitute the nonexpanding portions of stevensite, a method was sought that would differentiate between talc and other layer type structures that might also have a *c* axis spacing of 10 ~Å. As talc seemingly alone among the layer lattice silicates gives an infrared absorption pattern with a strong absorption peak at a wavelength close to 15 microns, it was thought that any talc present in the samples studied might be revealed by the occurrence of this peak in the infrared spectra.

Robert G. Milkey of the U. S. Geological Survey kindly made infrared absorption analyses of talc, hectorite, ghassoulite, and Stevensite No. 4 as well as saponite, phlogopite, and pyrophyllite, close neighbors of talc

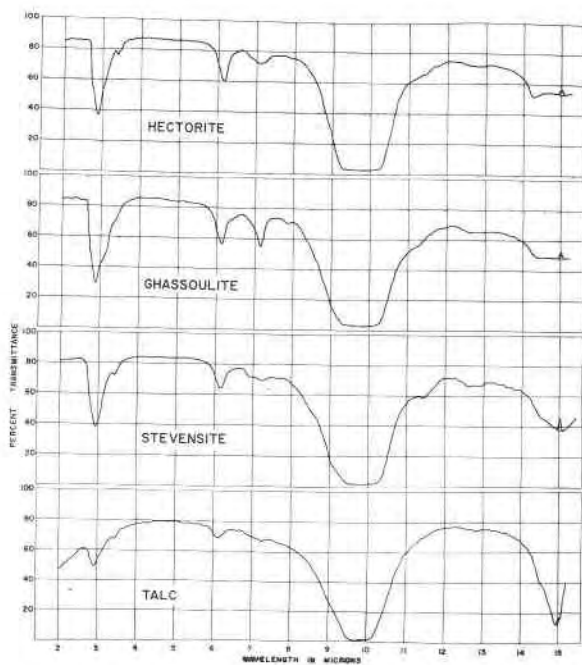


FIG. 5.—Infrared absorption spectra of hectorite, ghassoulite, stevensite and talc. Identical sample concentrations were used. The small sharp peak at 15 microns occurring in all the patterns is an instrumental effect. Robert G. Milkey, analyst.

in either composition or structure. The spectra obtained are shown in Figs. 5 and 6. Milkey's comments follow:

"Each of the minerals showed a strong absorption region at wave length 9 to 10 microns, which is shown by silicates in general. This peak, although showing some variations in shape among the different samples, was not distinctive enough to be useful for analysis of the structures.

"Other major absorption was due to hydroxyl and to the water molecules. These peaks are not necessarily quantitative, because no particular care was taken during the analysis to prevent absorption of water by the sample. It is of interest, however, that the hydroxyl peaks showed evidence of some non-hydrogen-bonded hydroxyl, together with stronger absorptions due to hydrogen-bonded hydroxyl which was largely accounted for as being present as H_2O molecules.

"The region of primary interest is at wave lengths from 14 to 15 microns. Talc has a strong absorption peak in this region at close to 15 microns, plus a weaker absorption peak near 14.5 microns. It seems likely that in this region, the absorption arises from the overall skeletal vibra-

tions of the crystal structure. Of the close neighbors of talc—stevensite, ghassoulite, saponite, hectorite, pyrophyllite, and phlogopite, only stevensite has a peak at this same point which is similar to that of talc. The intensity of the peak is somewhat less in the stevensite spectrum than in the talc. The other minerals showed only a diffuse absorption in this region: ghassoulite and hectorite showing a secondary peak near 14.4 microns, followed by a plateau of absorption, whereas saponite, pyrophyllite, and phlogopite had generally the diffuse plateau in this region.

“Spectra were also obtained of talc, and stevensite, ghassoulite, and hectorite which had been heated at 500° C. for 1 hour to eliminate inter-layer water and collapse the layers (Fig. 7). The heated sample of stevensite still showed, near 15 microns, the strong absorption peak, which now more closely resembled the talc peak than before. The ghassoulite and hectorite again did not show the strong peak at 15 microns; also, the minor peak which had previously appeared near 14.4 microns for ghassoulite and hectorite, was shifted after the layers were collapsed to a wave length of 14.5 microns. (It is interesting to note that this peak also occurs, as a weak ‘shoulder,’ in the spectra of stevensite and talc. In fact, the spectra of talc and stevensite in this region could possibly

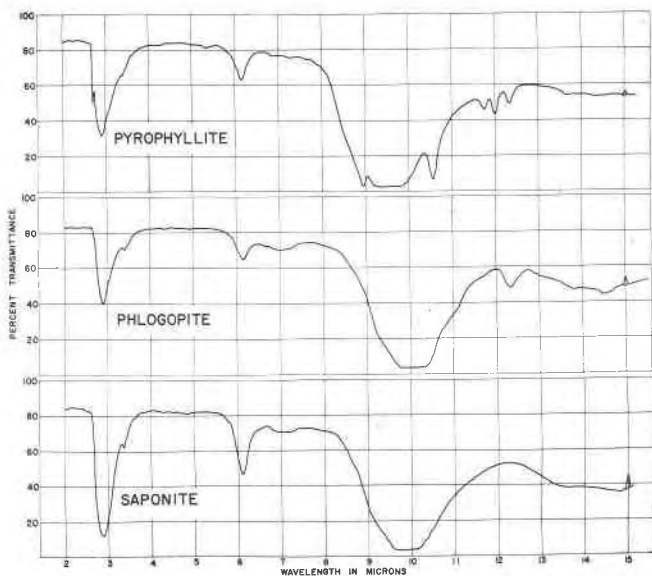


FIG. 6.—Infrared absorption spectra of pyrophyllite, phlogopite, and saponite. Identical sample concentrations were used. The small sharp peak at 15 microns occurring in all the patterns is an instrumental effect. Robert G. Milkey, analyst.

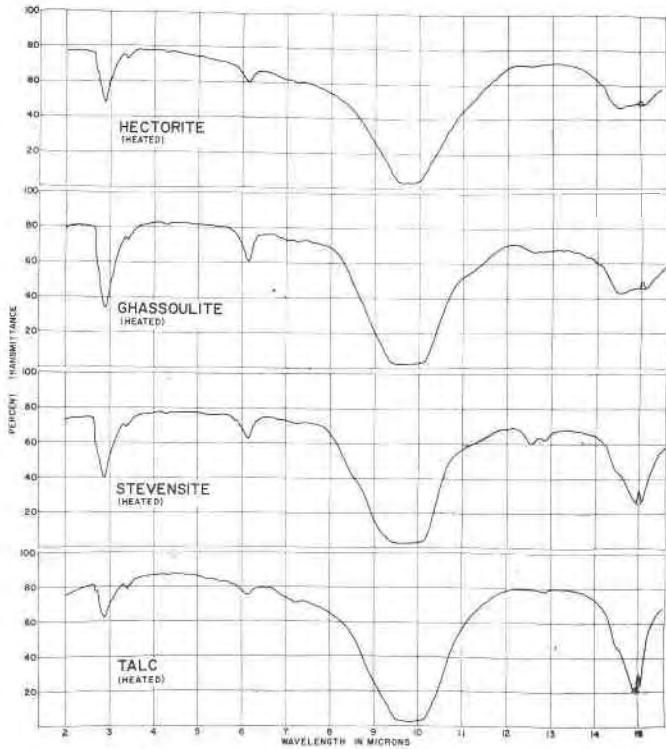


FIG. 7.—Infrared absorption spectra of hectorite, ghassoulite, stevensite and talc after each sample had been heated at 500° C. for one hour. Identical sample concentrations were used. The small peak at 15 microns occurring in all the patterns is an instrumental effect. Robert G. Milkey, analyst.

be the same as ghassoulite and hectorite, with the addition of the superimposed strong absorption at 15 microns.)”

On the basis of the infrared studies the data presented here do not unequivocally answer the questions regarding the presence of talc layers in the structures of stevensite. As Milkey states, “the particular type of bonding giving rise to the absorption in the 14.5 to 15 micron region is not known.” In addition the possible effect of the substitution of fluorine for hydroxyl in hectorite and ghassoulite has not been evaluated.

The significance of the infrared data for the heated and unheated specimens of talc and stevensite cannot be quantitatively assessed because the packing density was not determined and other quantitative factors were not controlled.

Bearing in mind these limitations, one could interpret the infrared data as suggesting that if non-expanding layers are present in stevensite they could be talc-like in nature.

SOLUTION STUDIES OF STEVENSITE, HECTORITE AND TALC

The ease of decomposing stevensite, and the other minerals now known to be identical with it, in hydrochloric acid has been known for a long time.

Samples of stevensite ground to the customary grain size for chemical analysis (-100 mesh) are completely destroyed by hydrochloric acid. Samples of talc similarly treated are not appreciably attacked. Murata¹ decomposed stevensite by leaching the mineral three times with 1:1 HCl. A sandy residue was formed. This residue, separated silica, is optically isotropic, has an index of refraction, n , of $1.45 \pm$ and is soluble in five per cent sodium carbonate solution.

Clarke and Schneider (1890) studied the solution of talc under the following experimental conditions—(1) heated in dry HCl gas to 383° – 412° C. for fifteen hours, (2) rapid evaporation with 75 cc. of fuming aqueous HCl, (3) eight days' digestion on the water bath with acid of 1.12 sp. gr., and (4) by digesting in like manner to (3) for thirty-two days. In the later experiment "3.94 (%) MgO and 0.41 (%) of sesquioxides were taken out." They conclude "Talc, therefore is, as should have been expected, remarkably stable in presence of hydrochloric acid, both aqueous and dry."

The marked contrast in the behavior of talc and stevensite under these experimental conditions is apparent.

Further data on the solution phenomena of stevensite, hectorite, and talc were obtained in this study.

The solution phenomena of stevensite were determined by digestion of the material in hot 10N HCl for about 6 hours. Two samples of stevensite were used and are referred to in the discussion as "1st run" and "2nd run." Both of these samples were prepared by gentle crushing, centrifuging and decantation. Mechanical deformation of the stevensite crystal plates has thus been kept at a minimum and the disorder in the crystal structure of the plates is not appreciably increased. Hectorite was likewise prepared and treated with acid. The results of these solution studies are recorded in Table 5. The column entitled "Wt. Loss in %, of original weight," in Table 5, includes not only the loss due to solution in the acid but also mechanical losses arising in filtering extremely fine-grained material which fails to settle or passes through the filter. This mechanical loss of sample gives rise to the discrepancy between the "MgO in %, of original wt." and the "MgO in typical sample of mineral."

The data for talc are not strictly comparable with those of stevensite for the samples were prepared by grinding dry for 30 hours in a mechanical mullite mortar. The starting material was a purified talc, U.S.P.,

¹ Murata, K. J., personal communication, 1953.

TABLE 5.—SOLUTION STUDIES OF STEVENSITE, HECTORITE, AND TALC IN HOT~10N HCl

Time of digestion in hours	Mineral	Wt. loss, in %, of original wt.	MgO in %, of wt. loss ¹	MgO in %, of original wt.	MgO in typical sample of mineral	Data on grain size	Notes
6	stevensite (1st run)	33.8	68.4	23.1	27.47	< 2 μ	stevensite No. 1 Springfield, N. J.
6	stevensite (2nd run)	32.5	62.5	20.3	27.47	< 2 μ	
6	hectorite	40.5	47.6	19.2	25.03	< 2 μ	Hector, Calif.
6	"ground talc" (no. T-1)			0.70	31.89	<37 μ	Original sample
6	"ground talc" (no. T-1)			7.6	31.89	10-20 μ	
6	"ground talc" (no. T-1)			10.5	31.89	2-10 μ	Original sample
6	"ground talc" (no. T-1)			11.1	31.89	1-2 μ	ground 30 hours
6	"ground talc" (no. T-1)			16.1	31.89	< 1 μ	
60	"ground talc" (no. T-1)			0.62	31.89	<37 μ	Original sample
60	"ground talc" (no. T-1)			8.5	31.89	10-20 μ	
60	"ground talc" (no. T-1)			10.8	31.89	2-10 μ	Original sample
60	"ground talc" (no. T-1)			12.3	31.89	1- 2 μ	ground 30 hours
60	"ground talc" (no. T-1)			17.0	31.89	< 1 μ	

¹ Harry C. Starkey, analyst.

Item 51-T-4350, from the Industrial Distributors which had already been ground to a fine grain size. A sample, no. T-1, ground for 30 hours give the following size analysis:

	<i>Weight per cent</i>
> 20 μ	1.6
10-20	7.8
2-10	38.0
1-2	10.5
< 1	36.3
	94.2
Experimental loss	5.8

This size analysis is unusual in having two pronounced modes. Some of this unusual distribution of the grain size may be due to the presence of incompletely ground particles of talc which remained at the periphery of the mortar during much of the grinding process. The various data on the solution phenomena of ground talc are given in Table 5. Determinations of the amount of Mg in the solutions obtained from stevensite and talc were made by Harry C. Starkey and John C. Hathaway of the U. S. Geological Survey using the automatic photometric titration method of Shapiro and Brannock (1956, p. 41).

In discussing solution phenomena and comparing the results of the various experiments it is necessary to define the conditions of the studies. The data discussed here are solution studies of these minerals in hydrochloric acid and are not solubility measurements. From the standpoint of thermodynamics and the phase rule a given point on the solubility curve, at a given temperature, represents an equilibrium relationship involving the coexistence of the liquid phase and the solid phase. Such a relationship cannot be established in acid solution studies.

In such inequilibrium processes external characteristics such as grain size, perfection of the crystals or crystalline particles and other properties may greatly influence the rate of, or amount of solution.

Minerals of the montmorillonite group such as stevensite are generally composed of aggregates of tiny plates. When such an aggregate is placed in water or an aqueous solution it will break up and swell. In swelling the plates of stevensite will separate parallel to the basal plane and become smaller than they were in the original mass. In the processes of dispersion, centrifuging and decantation the original tiny plates have not only become thinner but their other dimensions may be reduced. In addition, the stevensite in water may undergo some ion-exchange with hydronium ions. That these tiny plates may come from larger plates is indicated by optical examination of stevensite which shows double refracting areas. These doubly refracting areas in stevensite represent optical continuity over areas sometimes as much as a few square millimeters.

Talc, on the other hand, has strong cohesive forces which prevent its mechanical disintegration by water and in order to reduce it to comparable grain size with that of stevensite it is necessary to grind it. The continuous dry grinding of this talc has probably introduced considerable disorder into the crystal structure of the individual plates of talc. Such disordered crystals would be expected to have a greater ease of decomposition than (1) plates slacked off larger plates and (2) synthetic crystals of the same size. Yoder and Eugster (1955, p. 256-257) have shown that for muscovite, grinding under almost identical conditions for thirty hours not only affects the particle size but also the shape and that measurable structural damage was observed.

Synthetic talc crystals grown as aggregates in hydrothermal synthesis studies by Bowen and Tuttle (1949) were examined by differential thermal analysis and x -rays and found to consist only of talc. This material was also examined with the electron microscope by Marie L. Lindberg, U. S. Geological Survey, who found the fully dispersed material to be uniformly thin tabular plates. The range in size of the fully dispersed particles is from about $.02\mu$ to 0.26μ . The size of the plates was judged by

reference to included spheres of polystyrene having a diameter of 2590 angstroms. Solution studies on this synthetic talc were made by Joseph J. Fahey. The sample was treated with 10N HCl for six hours on the steam bath. Magnesium was determined as the pyrophosphate. The synthetic talc yielded 15.7% MgO in the solution study which indicates that it was about 50 per cent decomposed by the acid.

Bearing in mind all of these limitations we may conclude that stevensite aggregates are decomposed by hot 10N HCl at all aggregate grain sizes from -100 mesh to the $<2\mu$ dispersed particle fraction. Hectorite is likewise destroyed. Talc on the other hand is more resistant, it is not mechanically disaggregated by water or aqueous solutions, and for particles from -100 mesh down to at least 37 microns it is relatively unaffected by hydrochloric acid. For samples which have been ground dry for thirty hours the decomposition of the "ground talc" increases with a decrease in grain size and for the material $<1\mu$ approximately one-half of it is decomposed under these conditions. These various experiments confirm the relative stability of talc over stevensite in the presence of hydrochloric acid.

Infrared spectra of the residue from the stevensite, 1st run, and the talc acid treatments are shown in Fig. 8. The talc diagram is essentially unchanged from that of untreated talc, but the stevensite is considerably altered. A pattern similar to that of amorphous silica occurs and the 15 micron "talc" absorption is absent.

CATION EXCHANGE CAPACITY

The total cation exchange capacity of samples of the stevensite No. 4 from Paterson, N.J., ghaoulite, and hectorite were determined by Dorothy Carroll of the U. S. Geological Survey using the colorimetric manganese method of Bower and Truog (1940, p. 411-413). The values obtained are given in Table 6.

The total cation exchange capacities reveal differences between the three minerals, ghaoulite and hectorite falling within the range usually associated with the montmorillonite group minerals, and stevensite falling below the range. This is in accord with the previous studies reported by Faust and Murata (1953). Talc has a cation exchange of 0.2 to 0.4 m.e. per 100 grams Kingery, Halden, and Kurkjian (1955).

The exchangeable ions in ghaoulite were determined by Harry C. Starkey and John C. Hathaway using the flame photometric and automatic photometric titration methods of Shapiro and Brannock (1956, p. 39-44). The results are given in Table 7.

These results show that exchangeable Mg plays an important role in ghaoulite and that the sum of octahedrally coordinated cations is probably less than 3.

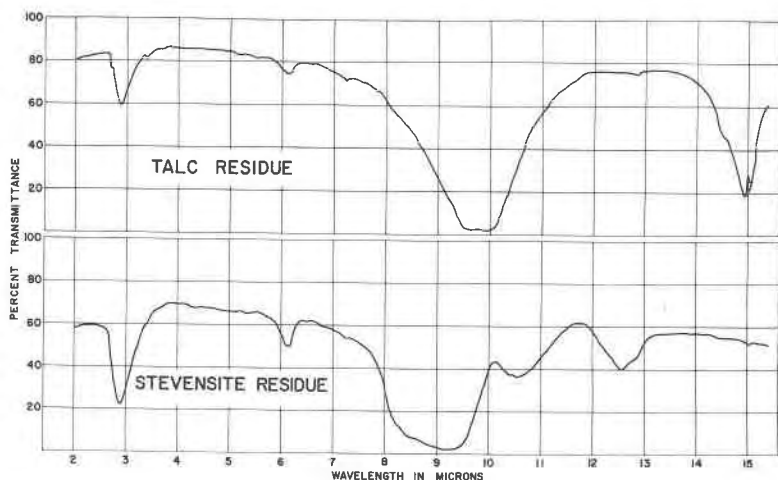


FIG. 8.—Infrared absorption spectra of residues from treatment of talc and stevensite with concentrated HCl. Identical sample concentrations were used. The small sharp peak at 15 microns occurring in all the patterns is an instrumental effect. Robert G. Milkey, analyst.

CHEMICAL ANALYSIS OF GHASSOULITE

Ghassoulite, analyzed by Augustin A. Damour in 1843 (Analysis 5, Table 1), contained 1.5% sand. The availability of ghassoulite from the studies of Millot (1954) afforded material for a new analysis. The new chemical analysis of ghassoulite, analysis No. 4 in Table 1, was made by our colleague, Joseph J. Fahey. Since the iron is chiefly in the ferrous state all of the iron is reported as FeO. The specific gravity of ghassoulite determined with a fused silica Adams-Johnston pycnometer using water as the filling liquid was found to be 2.336 at 4° C. The alkalis were determined by Joseph L. Dinnin using a flame photometer and the fluorine was determined by Sarah Berthold using a modified Willard-Winter method. It is of interest to note the general similarity between the

TABLE 6. TOTAL CATION EXCHANGE CAPACITY OF STEVENSITE, GHASSOULITE, AND HECTORITE

Mineral	Cation exchange capacity m.e./100 g.
Stevensite, No. 4, Paterson, N. J. <2 μ fraction	37.6
Ghassoulite, Morocco	75. ¹
Hectorite, California	60.8 ²

¹ Average of three determinations.

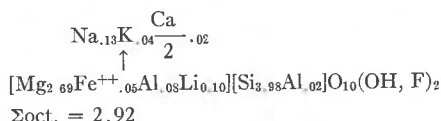
² Average of two determinations.

TABLE 7. CATION EXCHANGE RELATIONS OF GHAUSSOLITE

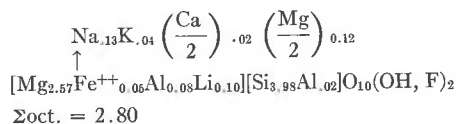
Exchangeable Cations	In m.e./100 g.	In per cent as oxides	
Na ⁺	17	Na ₂ O	.53
K ⁺	3	K ₂ O	.14
Ca ⁺⁺	2	CaO	.06
Mg ⁺⁺	55	MgO	1.13
Total	77		
Exchange capacity (from table 6)	75.1		

analyses of Damour and Fahey (note that 0.70% FeO \approx 0.78% Fe₂O₃).

To calculate the formula of ghassoulite 0.38% CaO was deducted to form with 0.30% CO₂, an amount of calcite equal to 0.68%. This gives 0.16% CaO as the amount in cation-exchange sites. Using the corrected analysis the formula is usually computed in the following form:



This formula must be modified however in the light of evidence given in Table 7 where Mg⁺⁺ is shown to be the principal cation-exchange ion. Using the measured cation-exchange for magnesium, one obtains the formula



The sum of 2.80 for the octahedrally coordinated ions is below that of either hectorite (2.98) or stevensite (2.86) and the charge on the structure is higher than in either of them.

INTERPRETATION OF THE DATA

In this study we have endeavoured to (1) find the relation between stevensite, ghassoulite, and hectorite and (2) to investigate further the interpretations of Brindley that stevensite is a talc-saponite interlayered mineral. All the methods we have used are not of the same sensitivity and for some techniques the results are not conclusive. It is desirable, therefore, to briefly summarize the results of each technique and to draw some conclusions from our research.

Differential thermal analysis studies

The DTA studies support the conclusions that stevensite, ghassoulite, and hectorite are all members of the montmorillonite group. The DTA curves for ghassoulite and hectorite up to 900° C. are more closely related to one another than to stevensite. Stevensite does not show a high temperature endothermic trough in the temperature range of 885° C. to 1061° C. where the dehydration of talc generally takes place. The absence of the high temperature endothermic trough, characteristic of talc, suggests that even if non-expanded layers are present in stevensite they are unlike talc.

Ghassoulite is also unlike talc in its behavior in DTA studies. Hectorite shows an endothermic trough beginning at 1053° C. extending as far as the record goes up to 1077° C. The significance of this trough has not been determined.

X-ray studies

The *x*-ray data for stevensite, ghassoulite, and hectorite reveal few differences between them other than the presence of a long spacing of about 25 Å in stevensite alone. As Brindley (1955, p. 242) has pointed out such a spacing could be explained on the basis of interstratification of 9.5–10 Å and 14–15 Å. Ethylene glycol treated oriented aggregates, however, gave patterns of essentially the same character for all of the minerals. The number and perfection of the basal reflections of hectorite and ghassoulite were little better than those of stevensite and all failed to show a strictly integral sequence of basal orders. Although the air-dried specimens of stevensite appeared to contain a large proportion of ~10 Å layers, less than 30% of non-expanded material would seem to be present in the glycol treated samples, otherwise a lower first order spacing would be expected.

The *x*-ray diffraction data thus give somewhat ambiguous results as to the differences between hectorite, ghassoulite, and stevensite.

Ghassoulite shows as little evidence of mixed layering as hectorite and like it exhibits no indication of "talc-like" layers upon collapse of the structure with heat treatment.

Infrared studies

Infrared absorption data on the untreated minerals show a distinct difference between stevensite and the other two minerals by the occurrence of the 15 μ "talc" absorption. The increase in intensity of this peak on heat treatment indicates that if non-expanded portions are present in stevensite, this phenomenon could be associated with such portions but it

is also to be noted that the expanding portions are capable of producing this same absorption after they are collapsed by heat treatment. The loss of this absorption peak from the stevensite pattern but not the talc pattern after hot concentrated HCl treatment suggests that although the non-expanded portions exhibit some of the characteristics of talc, they are sufficiently different to be destroyed by acid treatment.

Chemical studies

Solution studies on talc, stevensite, and hectorite show the unstable nature of stevensite and hectorite in concentrated acid solutions, a characteristic of montmorillonite group minerals, whereas talc is quite resistant under these conditions.

Cation exchange studies on stevensite, ghassoulite, and hectorite show that ghassoulite and hectorite are most closely allied in that they exhibit a much higher cation exchange capacity than stevensite.

Chemical analyses of stevensite, ghassoulite, and hectorite show that ghassoulite and hectorite are more closely related to one another than to stevensite in that they both have significant amounts of fluorine and lithium. Moreover, they both contain some alumina.

We come now to the terminology of Brindley in referring to stevensite as a "talc-saponite interlayered mineral." The usage of saponite by Brindley to apply to a pure magnesium montmorillonite is not in agreement with established usage. Saponite was originally defined by Svanberg in 1840 as a hydrous silicate of magnesium and aluminum. All subsequent usage of the name has been in accord with this definition. Further, substitution of Al for Si in tetrahedral coordination is considered by Ross and Hendricks (1945, p. 47) to be a characteristic of saponite. The one closely allied mineral containing essentially no aluminum (0.13)% discussed by Ross and Hendricks was hectorite and although they group it with other Mg-rich trioctahedral montmorillonites they do not include hectorite under the term saponite. Inasmuch as the analyses of stevensite and synthetic stevensite show no alumina, by established usage, no part of stevensite can be considered saponite.

After the completion of this paper we received an offprint by Caillère and Henin (1956) on the subject of the nomenclature of saponite, aphrodite, rassoulite, and stevensite in which they conclude (translated from the French):

"It seems now advantageous to preserve two names to designate the extreme types of this mineral group: one corresponding to a large substitution, the other to a very small substitution in the tetrahedral layer. In this case, one may retain respectively the end members saponite and stevensite."

Genesis of ghassoulite

It is noteworthy that all of the specimens of stevensite and hectorite are of hydrothermal origin. On the contrary the "earth of ghassoul" is a sedimentary facies found in beds between the flint horizon and the marly limestone horizon. This authigenic mineral formed in a sedimentary basin under conditions analogous to those described by Millot, Radier, and Bonifas (1957). The great mineralogical similitude of hectorite and ghassoulite does not prevent their having different origins.

CONCLUSIONS

The results of the present research on stevensite and related minerals do not confirm Brindley's concept of stevensite as a talc-saponite inter-layered mineral and indicate that stevensite is a type of defect structure. From the cation exchange studies, Faust and Murata (1953) deduced that a charge deficiency apparently originates in stevensite through a deficiency in the total number of ions in octahedral coordination. Any portion of the stevensite structure which has such deficiency has the attributes of montmorillonite and any portion of the structure of stevensite which does not have a deficiency in the octahedral layers has the attributes of talc. Since the deficiency in the octahedral layers of stevensite is so great, stevensite is a montmorillonite group mineral. A random, or statistically disordered, distribution of the vacant sites in the sheets of octahedrally coordinated atoms would give a small proportion of domains with few or no vacancies and which have the attributes of talc. The size of such domains in stevensite is not known. Our experimental data indicate that such domains in the structure of stevensite which would have the attributes of talc occur in units too small to scatter x -rays coherently. Bradley (1950) has shown that the diffraction effects observed in this study can be expected to be produced by the type of assembly proposed above.

This concept of stevensite as a defect structure seems to fit all the experimental data as follows:

- (1) The presence of domains with the attributes of talc in combinations with more abundant domains of stevensite could give rise to the weak reflection at 24.5–26.5 Å (\pm).
- (2) The appearance of the infrared absorption peak near 14.5 microns in natural stevensite and its disappearance in the residue of acid treated stevensite.
- (3) The absence of an endothermic trough corresponding to talc in the stevensite DTA pattern.
- (4) The complete decomposition of stevensite in acid.

The alternative explanation of Brindley that stevensite is made up of discrete interlayers of talc in saponite could be used to explain the occurrence of a weak reflection at 24.5–26.5 Å but it fails to explain observations 2, 3, and 4.

Theoretically one could derive the defect structure of the trioctahedral montmorillonite stevensite by creating, in random distribution, the maximum number of vacancies in the octahedral sheet (brucite sheet) in the talc structure and still preserve a stable crystal structure. Analogously, one could derive the defect structure of the dioctahedral montmorillonite by creating in random distribution the maximum number of vacancies in the octahedral sheet (gibbsite sheet) in the pyrophyllite structure. Rustum Roy and L. B. Sand (1956) have claimed the synthesis of this "defect pyrophyllite" structure which they called pure aluminum montmorillonite.

The defect structure theory proposed here is another way of deducing the dioctahedral and trioctahedral montmorillonite structures using as prototypes-talc and pyrophyllite. The original usage of talc and pyrophyllite as prototypes for montmorillonites was proposed by Hofmann, Endell and Wilm (1933) and modified by Marshall (1935) and Hendricks (1942). This theory deduces the structure of montmorillonite from the structure of pyrophyllite and talc through the replacement of silicon in tetrahedral coordination by trivalent aluminum, or by the replacement of aluminum in pyrophyllite or magnesium in talc by other ions, or by a combination of both types of replacements.

Vacant sites in the trioctahedral montmorillonite-sauconite have been discussed by Faust and Murata (1953).

RELATIONSHIPS AMONGST STEVENSITE AND SOME SIMILAR MINERALS OR MIXTURES

In addition to the minerals studied by us, other investigators have reported on related minerals, or on the synthesis of hydrous magnesium silicates which are discussed below in the light of our own studies.

Syntheses of stevensite

Strese and Hofmann (1941) were probably the first to synthesize stevensite. Their preparation "Synthetischer Magnesium montmorillonit 33B" gives an x-ray pattern comparable with that of natural hectorite. Their experimental data suggest that for most of their preparations K^+ made up most if not all of the cation exchangeable ions; in some it was Ca^{++} and probably also Mg^{++} .

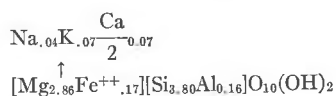
Bowen and Tuttle (1949) in their study of the system $MgO-SiO_2-H_2O$ attempted to synthesize the mineral sepiolite. They were apparently not

entirely satisfied with the identification. They based their preparation on the formula $2\text{MgO} \cdot 3\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ which differs from stevensite in having about 3% more silica and about 3% less water. They mention (p. 441) that the experimental charges lost silica by transport in the vapor phase and accordingly the formation of stevensite in their experiment is not unexpected. X-ray and DTA studies of a large sample prepared by them for Faust and reported on in this paper shows the charge to consist of stevensite with a very small amount of chrysotile.

Roy and Roy (1955) accomplished the synthesis of stevensite in the system $\text{MgO-SiO}_2\text{-H}_2\text{O}$ and Mumpton and Roy (1956) studied the stability temperature of this synthetic stevensite.

Interstratified "saponite-talc" of Alietti

Alietti (1956) described an interlayered saponite-talc mineral from Monte Chiaro, Italy and his chemical analysis is given in Table 1. The original paper contained a misprint for the value of silica of 5.31, which was corrected in ink to 54.31. A recalculation of this analysis yielded the formula



which does not agree with that given by Alietti namely



There may be some error in the reporting of the data.

On the basis of our calculations there is no deficiency in the octahedral layer ($\Sigma = 3.03$) and this mineral could have no stevensite layers in its structure. However, as we showed in the section on the formula of ghas-soulite, cation exchangeable magnesium must be determined on minerals of this kind in order to determine the actual composition of the octahedral layer. Until this is determined one cannot use Alietti's chemical analysis to establish the presence and proportions of stevensite-type or saponite-type layers in the structure. An examination of Alietti's other data does confirm his interpretations that the mineral he studies is a montmorillonite-type with regularly interstratified non-expanding layers, in the proportion of 1:1 as judged by his diffractometer patterns, 1b and 1c (p. 203).

Hanušite

Kašpar (1942) studied a hydrous magnesium silicate which he called hanušite. Hanušite occurs as yellowish-white to yellow-brown radiating aggregates, similar to pectolite, in the tholeiite of the Liebstadt region

in the Riesengebirge. Kašpar concluded that hanušite was the magnesium end member in the supposed series pectolite-walkerite. Kašpar was unaware of the work by Glenn (1916), or he would have recognized that stevensite had already been assigned to the name of the magnesium end member of this supposed series. An examination of Kašpar's data leaves little doubt that hanušite is a mixture of stevensite and pectolite. Thus, there is no alumina in hanušite, it is completely decomposed by HCl, the dehydration curve is similar to that of montmorillonites and the chemical composition after subtraction of hypothetical pectolite from the analysis yields a composition of the right order of magnitude for stevensite. Using the CaO value in Kašpar's analysis and Fig. 1 in Faust and Murata (1953) hanušite appears to be made up of about 88% stevensite and 12% pectolite.

RECOMMENDATION

It is recommended that the names hanušite (= stevensite+pectolite) and ghassoulite (= hectorite) be relegated to the synonymy.

ACKNOWLEDGMENTS

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