

The Crystal Chemistry of Epistilbites

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Abstract

The zeolite, epistilbite, occurs primarily as a low temperature-alteration product of basic igneous rocks. Chemical analyses, crystallographic data, and densities were obtained for 13 specimens. The chemical composition varies within narrow limits from the approximate composition $\text{CaAl}_2\text{Si}_8\text{O}_{18} \cdot 5\text{H}_2\text{O}$. Tetrahedral Si varies between 72.5 and 77.2 wt percent. Ca dominates the exchangeable cations, and K is always scarce or absent. The cell constants also vary within narrow limits: $a = 9.089\text{-}9.102$, $b = 17.741\text{-}17.802$, $c = 10.205\text{-}10.242$ Å, and $\beta = 124.55\text{-}124.68^\circ$. The measured density varies within 2.22-2.28 g cm⁻³. The largest correlation (negative) between chemical and crystallographic data occurs between (Ca + Ba) and β . Smaller correlations occur between a and c (positive) and between Si/(Si + Al) and (Ca + Ba) (negative). The DTA curves of 12 of the samples can be divided into groups of differing complexity. A listing of the occurrences reported in the literature is given.

Introduction

Epistilbite, described as a new mineral by Rose (1826), is a rather common but often misidentified zeolite. Names synonymous with epistilbite but now obsolete are: *monophane* (Breithaupt, 1823), *parastilbite* (Von Waltershausen, 1853), *reissite* (Von Fritzsche, 1870), and *orizite* (Grattarola, 1879). Its structure was described first by Kerr (1964), and confirmed by Merlino (1965), Perrotta (1967), and Slaughter and Kane (1969).

We now establish the range of chemical variation of epistilbite and the relationships between chemical composition and physical properties.

E. Galli was responsible for the sample collection. X-ray diffraction work, density determination, TG and DT analyses; and R. Rinaldi for the electron microprobe analyses.

Description of the Samples

The samples are listed in Table 1. The most common crystal habit for our epistilbites is that of Figures 15 and 28 of Tables 141, 143 in Goldschmidt (1916) Band B (Tafeln). Other habits were like those of Figures 2, 17, 21, 23. A different crystal habit is that of sample no. 1 (Elba Island, Italy—"orizite")

whose crystals are often grouped in cross-shaped or irregular aggregates: their shape, luster and color are reminiscent of rice grains. In sample no. 12 (Furuyada, Japan) no crystal faces are recognizable since the mineral fills completely the cavities in the host rock. The true origin of sample no. 5 (Castle Eden, England) is unknown (P. G. Embrey, British Museum, personal communication). It was collected by the late Dr. C. O. Trechmann, who lived at Castle Eden but collected the samples from a nearby pile of basalt fragments used as road material.

We list in Table 2 all other localities where epistilbite has been inferred to occur, in order to give as complete a list as possible of occurrences and to emphasize that the presence of epistilbite in several of these localities is doubtful or unacceptable. We tried to obtain samples from many other localities (listed in Table 2), but usually we were informed that the samples were not available in the museums or in nature.

No sedimentary epistilbite has been reported to occur in nature.

Experimental

Electron microprobe analyses for Si, Al, Ca, Ba, Na, and K were carried out using an ARL instrument

TABLE 1. List of Samples Herein Studied, with Their Occurrences

No.	Locality of occurrence	Habit ^a , paragenesis, and note	Reference	Donor (if not owned by Modena University)
1	Fonte del Prete, near San Piero in Campo (Isola d'Elba, Italy)	Vitreous translucent prismatic crystals (up to 2x1.5x3 mm), usually intergrown and twinned, recalling the shape of rice grains. Note: also called orizite.	Merlino (1972) Grattarola (1879)	University of Pisa; No. 53
2	Giebelbach, near Fiesch (Valais, Switzerland)	Aggregates of white transparent vitreous crystals (up to 1.5x0.5x2 mm). Habit 15 and 28.	Hintze (1897)	University of Bologna
3	Finkenhübel, near Glatz [=Kłodzko], Silesia (ex-Germany, now Poland)	White transparent crystals in cavities of amygdaloidal rock.	Hintze (1897)	University of Turin; No. 10380
4	Madap, Velence hills (Com. Fejér, Hungary)	Small vitreous crystals, perfectly transparent, with Stilbite, sphalerite and pyrite. Habit 2.	Mauritz (1908)	Natural History Museum, Budapest
5	Castle Eden, near Hartlepool (Durham Co., England)	Aggregates of small (up to 1.5x0.6x2 mm) vitreous transparent crystals completely filling the cavities in basalt. Habit 17.	Hintze (1897)	British Museum (Natural History): part BM 1917,722
6	Djupivogur [3 km from the Mt. Bulandstindr], (Berufjörd, Iceland)	Large (up to 7x3.5x10 mm) colorless translucent crystals, intergrown and twinned, forming irregular tabular laths. Habit 28.	Hintze (1897)	
7	Berufjörd, at the foot of the Mt. Bulandstindr (Iceland)	Aggregates of colorless, cloudy or reddish crystals on black oily rock.	Hintze (1897)	University of Turin; No. 9836
8	Theigarhorn [=Tiegarnhorn], (Iceland)	Aggregates of large (up to 8x2x10 mm) colorless, cloudy or transparent crystals, complexly intergrown, and forming irregular roof-shaped twins, with mordenite on reddish igneous rock. Habit 21.	Slaughter and Kane (1969)	University of Turin; No. 13581
9	East-side of øfjörd (Iceland)	Small vitreous transparent crystals in cavities in basalt.		Mineralogical Museum (University of Copenhagen)
10	Farøe islands	Large (up to 4x1.2x6 mm) white translucent crystals, complexly intergrown and twinned. Habit 23.	Hintze (1897)	Mineralogical Museum (University of Copenhagen)
11	Yugawara (Kanagawa Pref., Japan)	White transparent crystals (up to 2x0.6x2.5 mm), with quartz in cavities in igneous rock. Habit 15 and 28.	Koizumi (1953)	National Science Museum of Tokyo, Japan
12	Furuyada, Mitama (Yamanashi Pref., Japan)	Milky white crystals, with heulandite and pumpellyite aggregate on epistilbite completely filling the cavities in basalt.		National Science Museum of Tokyo, Japan
13	Kumomi, Matsuzoki (Shizuoka Pref., Japan)	Small vitreous transparent crystals, with chabazite and quartz on igneous rock. Habit 15 and 28.		National Science Museum of Tokyo, Japan

^aThe number of the habit refers to the number of the figures in Tables 141-143 in Band III (Tafeln) of Goldschmidt (1916).

operated at 10 kV, 0.1 μ A beam current and approximately 10 μ m spot size in order to avoid damage to or disintegration of the sample. These conditions were selected after testing various settings of the instrument for loss of Na using one zeolite grain mounted for this purpose. Ten readings for each sample were taken with a counting time of 10 seconds each. An₅₀ synthetic plagioclase glass was used as a standard for Si, Al, Ca, and Na; Kokomo sanidine for K and Ba. The data was reduced following the "Bence-Albee" method (Bence and Albee, 1968; Albee and Ray, 1970), and by the computer program EMPADR VII (Rucklidge and Gasparrini, 1969, Dept. Geology, University of Toronto) modified by Steele (1973, Dept. of Geophysical Sciences, University of Chicago).

Water loss was determined on approximately 10 mg of material by means of TG analysis using a thermal analyzer manufactured by B.D.L. (Bureau de Liaison, Paris). DTA analyses were also executed on a B.D.L. instrument; heating rate of 10°C per minute, 6 μ l Pt sample vials, Δt attenuators at 200 V and Δw attenuators at 20 mV.

Density was measured using a torsion microbalance and toluene, according to Berman (1939).

The cell dimensions were derived by a least-squares refinement program applied to the diffraction data measured on a Philips powder diffractometer. The instrumental settings were: Ni-filtered $\text{CuK}\alpha_1$ radiation ($\lambda = 1.54051$) for $2\theta \geq 18^\circ$; 40 kV, 20 mA; angular speed $\frac{1}{4}^\circ 2\theta$ per minute, 0.5-0.1-0.5 slits for $2\theta < 20^\circ$ and 1-0.1-1 for $2\theta > 20^\circ$. Cubic $\text{Pb}(\text{NO}_3)_2$ with $a = 7.8568 \text{ \AA}$ (see 6-0151 JCPDS card) was used as internal standard. The cell dimensions were refined by (a) approximately determining these cell parameters from ~ 20 lines which could be unequivocally indexed and which were near standard lines; (b) calculating all d_{hkl} values from these approximate parameters; (c) indexing of the whole tracing, taking into account the observed structure amplitudes (Perrotta, 1965, Ph.D. Thesis, University of Chicago); (d) least-squares refinement of cell parameters using the lines indexed in step (c). For every sample, steps, (b), (c) and (d) were repeated at least three times, every time more lines (up to 40) were unequivocally indexed.

TABLE 2. List of Occurrences Reported in the Literature to Yield Epistilbite, But out of Reach for the Present Study

No.	Locality of occurrence, and note	Reference
1	Bay of Fundy, near Blomidon(Nova Scotia-CANADA)	Smith(1971)
2	Margaretville, near Port George(Annapolis Co., Nova Scotia-CANADA)	Hintze(1897)
3	Lohja(SW FINLAND)	Duhovnik(1949)
4	Gierwiese, near Honnef(Rheinpreussen-GERMANY). Note: identification uncertain.	Hintze(1897)
5	Greinfenstein, near Ehrenfriedersdorf and Schwarzenberg(Saxony-GERMANY)	Tetzner and Edelmann(1927)
6	Akrotiri(Santorin[=Thera]-GREECE). Note: also called Reissite.	Hintze(1897)
7	Sátoros(Com. Nógrád-HUNGARY)	Erdélyi(1942)
8	Thyrill, at the beach of Hvalfiord(Borgafiord-Iceland). Note: also called Parastilbite.	Hintze(1897)
9	Igatpuri(NE of Bombay-INDIA)	Hintze(1897)
10	Poona(INDIA)	Dana(1914)
11	Sirur, Ahmadnagar Dst.(Bombay-INDIA)	Hey(1932)
12	Palagonia(Sicilia-ITALY). Note: optical identification only.	Sturiale(1963)
13	Amagi(Izu Pref.-JAPAN)	Wada(1904)
14	Nikko and Ohora(JAPAN). Note: identification uncertain.	Jimbó(1899)
15	Also-Vácza(Transylvania-RUMANIA, Hungary at the time of the reference). Note: identification uncertain.	Hintze(1897)
16	Mt. Calvary, near Nagyág(Transylvania-RUMANIA, Hungary at the time of the reference)	Hintze(1897)
17	Lunddörrsfjall(SWEDEN). Note: identification uncertain.	Hintze(1897)
18	Dannemarche reservoir, St. Lawrence valley(Jersey-UK)	Hey and Mourant(1933)
19	Rathlin and Portrush(N. Ireland-UK)	Hintze(1897)
20	Skye island(Scotland-UK)	Hintze(1897)
21	Kibinsky and Lovozersky(USSR). Note: identification uncertain.	Fersman(1922)
22	Mt. Tzkhara-Tzkhara(Trans-Caucasia-USSR). Note: identification uncertain.	Smirnov(1924)
23	Baylis quarry, near Bedford(New York-USA)	Pough(1936)
24	Bergen Hill (New Jersey-USA). Note: wrong identification; the mineral is thomsonite according to Confield(1911).	
25	Danbury(Connecticut-USA)	Pawloski(1965)
26	Lanakai hills(Hawaii-USA)	Dunham(1933)

TABLE 3. Electron Microprobe and TGA Analyses, and Atomic Ratios of Epistilbite

	1	2	3	4	5	6	7	8	9	10	11	12	13
SiO ₂	61.9	57.8	57.2	59.5	56.9	57.0	60.5	58.0	61.5	58.7	57.4	58.7	59.7
Al ₂ O ₃	15.5	17.0	16.7	17.0	18.3	17.7	16.9	17.0	15.8	16.7	17.7	16.4	16.3
CaO	6.8	8.3	8.0	8.7	8.4	8.1	7.2	7.8	7.3	7.8	8.4	8.6	8.1
BaO	0.0	0.0	0.3	n.d.	0.0	0.0	0.1	0.0	n.d.	0.0	0.0	0.0	n.d.
Na ₂ O	1.3	0.5	0.6	0.7	1.1	1.6	1.9	1.5	1.5	1.0	0.6	0.5	0.5
K ₂ O	0.8	0.8	0.5	0.2	0.0	0.1	0.3	0.1	0.0	0.0	0.1	0.0	0.0
H ₂ O	14.5	15.3	15.2	15.5	16.0	15.1	15.2	15.3	15.2	16.1	15.3	15.6	16.0
Total	100.8	99.7	98.5	101.6	100.7	99.6	102.1	99.7	101.3	100.3	99.5	99.8	100.6
Si	18.6	17.8	17.9	17.9	17.5	17.6	18.1	17.9	18.4	18.0	17.7	18.1	18.2
Al	5.5	6.2	6.2	6.1	6.6	6.5	6.0	6.2	5.6	6.1	6.4	6.0	5.9
Ca	2.2	2.8	2.7	2.8	2.8	2.7	2.3	2.6	2.4	2.6	2.8	2.8	2.7
Ba	0.0	0.0	0.0	n.d.	0.0	0.0	0.0	0.0	n.d.	0.0	0.0	0.0	n.d.
Na	0.7	0.3	0.4	0.4	0.6	1.0	1.1	0.9	0.9	0.6	0.4	0.3	0.3
K	0.3	0.3	0.2	0.1	0.0	0.0	0.1	0.1	0.0	0.0	0.1	0.0	0.0
O	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0	48.0
H ₂ O	14.5	15.8	15.8	15.6	16.4	15.5	15.2	15.7	15.2	16.5	15.7	16.0	16.3
R ^a	0.772	0.742	0.744	0.748	0.725	0.731	0.752	0.742	0.768	0.749	0.733	0.752	0.756
E ^b	1.77	1.20	2.91	-1.13	6.60	1.40	2.10	1.97	0.21	5.63	7.34	-0.25	5.23

^aR = Si/(Si+Al).

^bE = Balance error (see Passaglia, 1970).

MgO and SrO were also tested and found absent in all the samples.

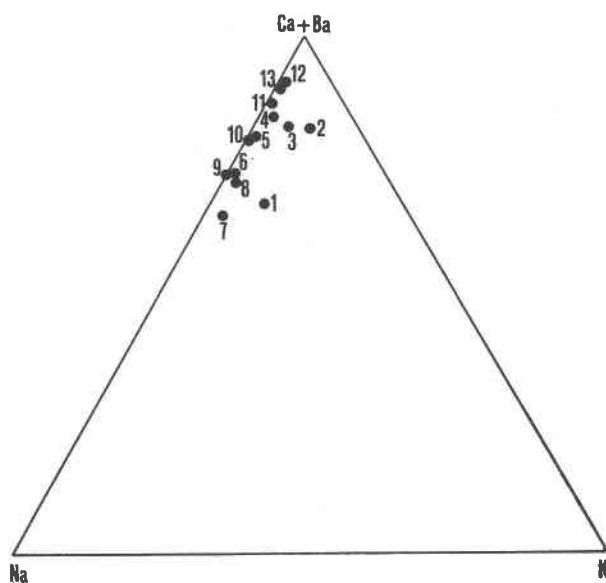


FIG. 1. Molar plot of exchangeable cations in analyzed epistilbites.

Results and Their Variability

The chemical analyses are reported on Table 3 with their balance error, $100 \times (Al_{obs} - Al_{theor}) / Al_{theor}$, where $Al_{theor} = Na + K + 2Ca$ (molar), (Passaglia, 1970). Mg and Sr were not detected in any sample. The variability of the exchangeable cation content

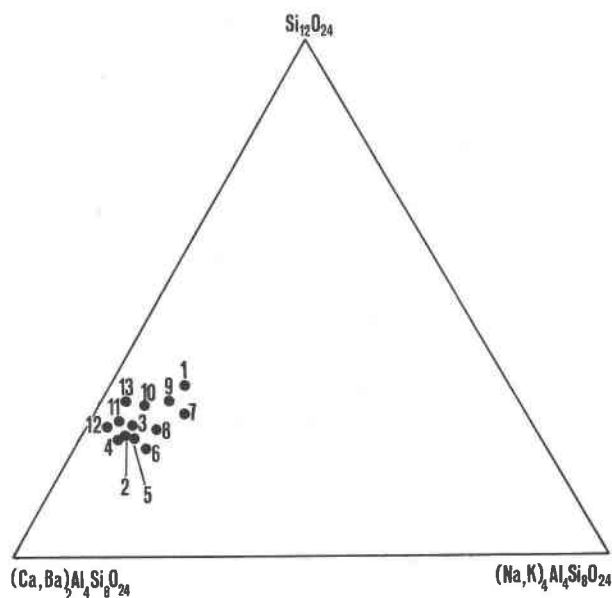


FIG. 2. Molar plot of $Si_{12}O_{24} - (Ca,Ba)_2Al_4Si_8O_{24} - (Na,K)_4Al_4Si_8O_{24}$ in analyzed epistilbites.

TABLE 4. Cell Parameters of Epistilbites

No.	a(Å)	b(Å)	c(Å)	β	v(Å ³)
1	9.088(1)	17.741(1)	10.225(1)	124.68(1)	1355.6(1)
2	9.089(1)	17.752(2)	10.226(1)	124.57(1)	1358.6(2)
3	9.087(1)	17.766(1)	10.220(1)	124.58(1)	1358.4(2)
4	9.102(1)	17.770(2)	10.241(1)	124.55(1)	1364.3(2)
5	9.096(1)	17.775(1)	10.229(1)	124.58(1)	1361.7(2)
6	9.098(1)	17.770(1)	10.240(1)	124.58(1)	1363.0(2)
7	9.088(1)	17.756(1)	10.236(1)	124.60(1)	1359.7(2)
8	9.094(1)	17.775(1)	10.235(1)	124.59(1)	1362.0(1)
9	9.088(2)	17.741(3)	10.238(2)	124.62(2)	1358.4(3)
10	9.095(1)	17.778(1)	10.220(1)	124.61(1)	1360.0(1)
11	9.096(1)	17.761(2)	10.236(1)	124.58(1)	1361.4(2)
12	9.082(1)	17.802(2)	10.205(1)	124.55(1)	1359.0(2)
13	9.101(1)	17.766(2)	10.242(1)	124.61(1)	1363.0(2)

is illustrated in Figure 1; the analyses are plotted in terms of molecular percentages of Ca + Ba, Na, and K. The diagram shows that the cationic composition of epistilbites varies very little; Ca always dominates the exchangeable cations and K is very low in almost all samples. The compositional range of SiO_2 and Al_2O_3 (Fig. 2) is limited to a very restricted area between 8.8 and 9.3 Si atoms per 12 tetrahedral sites. The tetrahedral Si/Al ratio in epistilbite is close to that of albite. The unit cell content can be described with fairly good approximation by $6\{Ca_{0.5}[AlSi_3O_8]2.5H_2O\}$.

The water content of epistilbite ranges only from 14.5 to 16.5 molecules per unit cell. The composition of epistilbite is very close to that of stilbite and is near that of some heulandites.

The lattice constants of the 13 samples are listed in Table 4. A very limited variation occurs in the lattice constants: $9.082 \leq a \leq 9.102$ Å, $17.741 \leq b \leq 17.802$ Å, $10.205 \leq c \leq 10.242$ Å, and $124.55 \leq \beta \leq 124.68^\circ$. The experimental error was rounded in the table to a minimum value of 0.001 Å, but was often 0.0005 Å or less.

Measured densities (2.22–2.58 g/cm³) and calculated densities agree well (Table 5).

DTA curves for samples no. 1, 7 and 11 (Fig. 3)

TABLE 5. Densities (g cm⁻³) of Epistilbites

No.	ρ (meas)	ρ (calc) ^a	No.	ρ (meas)	ρ (calc) ^a
1	2.22(1)	2.221	8	2.25(1)	2.247
2	2.25(2)	2.259	9	2.22(2)	2.228
3	2.25(1)	2.260	10	2.26(1)	2.262
4	2.23(1)	2.240	11	2.25(1)	2.247
5	2.28(1)	2.268	12	2.26(2)	2.254
6	2.26(1)	2.248	13	2.25(1)	2.245
7	2.23(1)	2.238			

^a ρ (calc) was obtained on the basis of 48 oxygens per unit cell.

show a gradual passage from one to the other of the three groups. Sample no. 1 shows only two endothermic peaks, the first one wider and deeper with its maximum at 150°C, the second one shallower with its maximum at 335°C. A very different behavior is shown by sample no. 11. Besides the two large endothermic peaks at 125 and 300°C, there is a flexure at 245°C and a third endothermic peak, less pronounced than the first two, at 340°C. The DTA curve of sample no. 7 shows an intermediate behavior. The flexure at 245°C is no longer present while the peak at 335°C is reduced to a flexure. The two peaks at 125 and 305°C are always present. All samples showed, after 900°C, the beginning of an exothermic reaction. The endothermic peaks result from expulsion of zeolitic water. The last exothermic peak can be explained with the beginning of recrystallization of the sample.

Differential thermal analysis was carried out for all samples except no. 9 for which not enough material was available. All curves obtained are similar to one of the three types reported in Figure 3. Sample no. 12 gave a curve similar to that of no. 1; no. 8 to that of no. 7, and nos. 2, 3, 4, 5, 6, 10, and 13 to that of no. 11. These differences in the DTA curves, among samples of very similar chemical composition and lattice parameters, could be due to some structural peculiarity such as stacking faults that cause a partial occlusion in the channels present in the zeolite framework. The presence of stacking faults in epistilbite has been emphasized also by Slaughter and Kane (1969).

The different modalities of water expulsion shown by the three samples of Figure 3 are confirmed by the corresponding TG analyses. The samples belonging to type 11 show the most complex stepwise dehydration phenomena. Koizumi (1953) found the same complexity in the dehydration curve of a sample of epistilbite from Yugawara (Japan), which is the same locality as for sample 11.

Correlation Between Chemical Composition and Unit Cell Parameters

A complete correlation analysis was carried out between the following variables: a , b , c , β , V , R , M , B , $(M + B)$ and H_2O where V = unit cell volume, $R = Si/(Si + Al)$, $M = Na + K$, $B = Ca + Ba$, and H_2O is the number of water molecules per unit cell. In the course of this analysis every variable was assumed in turn to be independent; the data in Tables 3 and 4 were tested for possible

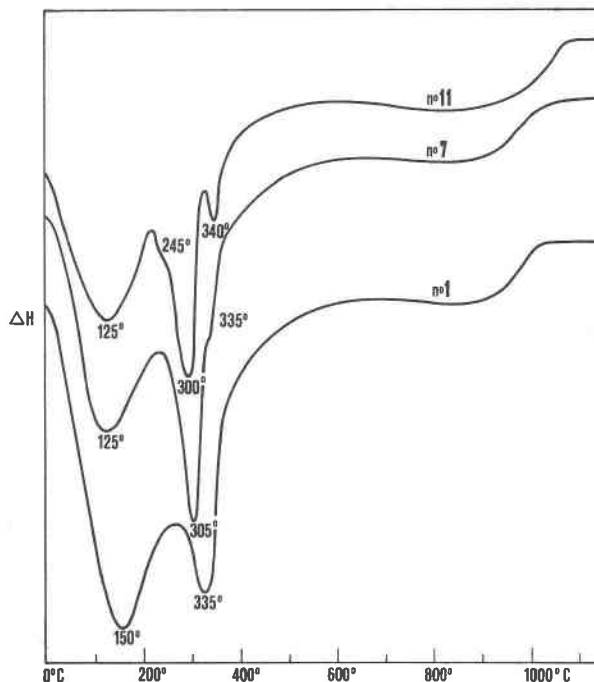


FIG. 3. DTA curve of epistilbite (heating rate: 10°C per minute).

correlations using the BMD 02R stepwise multiple-regression program (Sandi and Franchi, Centro Scientifico IBM, Pisa, Italy). The correlation matrix is reported in Table 6. Unexpectedly, the highest

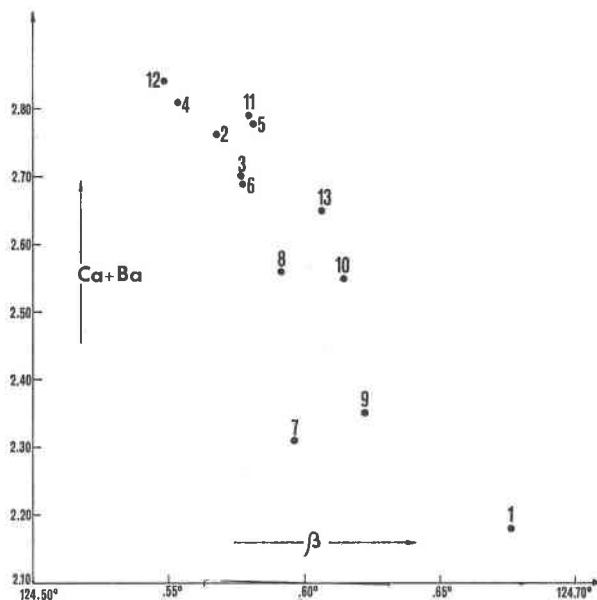


FIG. 4. Plot of $B (= Ca + Ba)$ versus β in the unit cell of epistilbite.

TABLE 6. Correlation Matrix between Cell and Chemical Parameters in Epistilbites

	a	b	c	β	V ^a	R ^b	M ^c	B ^d	(M+B)	M/(M+B)	H ₂ O
a	1.000	.050	.706	-.133	.881	-.386	-.182	.305	.063	-.218	.447
b		1.000	-.477	-.638	.321	-.437	-.481	.649	-.007	-.541	.544
c			1.000	.108	.647	-.086	.281	-.155	.251	.254	-.022
β				1.000	-.467	.645	.483	-.871	-.234	.573	-.358
V ^a					1.000	-.594	-.199	.499	.249	-.280	.513
R ^b						1.000	.171	-.709	-.523	.273	-.278
M ^c							1.000	-.744	.679	.992	-.604
B ^d								1.000	-.016	-.815	.482
(M+B)									1.000	.589	-.375
M/(M+B)										1.000	-.619
H ₂ O											1.000

^aV = unit cell volume.
^bR = Si/(Si+Al).
^cM = Na+K.
^dB = Ca+Ba.

(negative) correlation occurs between β and B (Fig. 4). The structural meaning of this is difficult to interpret. A negative correlation of R with a , b , c , or V was expected but does not occur. Lower correlations, but still of some significance, occur between a and c (positive) and between R and B (negative).

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