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X-RAY AND ELECTRON DIFFRACTION DATA FOR SEPIOLITE*

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ABSTRACT

X-ray powder data for sepiolite are indexed on the basis of an orthorhombic cell. The mineral shows marked variations in crystallinity. Single crystal, electron diffraction diagrams confirm the *b* and *c* parameters, and in conjunction with electron microscope observations prove that the lath-like crystals develop on the (100) plane.

X-RAY DATA

X-ray, dehydration, thermal, and other measurements by Caillère (1936), Migeon (1936) and Longchambon (1937) on sepiolites from various sources have established that sepiolite is a distinct mineral. The "X-Ray Powder Data File" (1958 Edition) contains data for four sepiolites additional to those given by the above investigators. There are very considerable differences among these data and none of them is indexed. Collectively they may suffice to identify the mineral, but they leave much uncertainty regarding the weaker x-ray reflections.

The crystal structure of sepiolite has been analyzed by Nagy and Bradley (1955), who showed that the mineral consists of mica-like units extending parallel to the fiber axis (*c*-axis); Fig. 3 shows schematically a cross-section of a sepiolite fiber based on their analysis. Nagy and Bradley indexed the *hk*0 reflections in an *x*-ray fiber diagram, but these do not account for all the reflections in a powder diagram. They showed that the unit cell is either orthorhombic or monoclinic and gave the cross-section of the cell as 13.4×27.0 Å. Preisinger (1957) stated that the cell is orthorhombic with parameters.

$$a = 13.4, \qquad b = 26.8, \qquad c = 5.26 \text{ Å}$$

in agreement with the results of Nagy and Bradley. His results have been published so far only in abstract form. The space group was suggested as probably $C2/m$ by Nagy and Bradley, and as $P2_1/n\ 2/c\ 2/n$ by Preisinger. On the basis of powder measurements it is scarcely possible to make reliable comments on the space group and it will be shown that the data can be indexed on the basis of an orthorhombic cell with $(h+k)$ even.

In the present work sepiolites have been examined from various localities including Little Cottonwood, Utah; Gouverneur, New York; Knights Quarry, Cal.; Cornwall, England; Vallecas, Spain; an unknown locality in Kenya; and Eski Chehir, Asia Minor.

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Most of these materials appear to be pure or almost pure sepiolite as judged by the absence of unusual reflections, but the crystallinity varies considerably. Extreme cases are illustrated in Fig. 1 which shows x-ray diffractometer records of sepiolites from Little Cottonwood, Utah (the upper trace), and from Eski Chehir (the lower trace). The lattice spacings and reflected intensities for these materials, and for two others of intermediate degree of crystallinity from Vallecas, Spain, and from Kenya, are listed in Table 1, where the d-values are measured with respect to CuKα radiation (λ1.5418 Å).

The powder data are indexed in terms of the following unit cell:

$$a = 13.50 \pm 0.02, \qquad b = 26.97 \pm 0.03, \qquad c = 5.25_6 \pm 0.01 \text{ Å}$$

The indices hkl and the calculated d values are given in Table 1, and are complete down to $d = 2.40$ Å. Additional calculated values down to $d = 2.0$ Å are given which correspond to observed reflections. For $d < 2.0$ Å the calculated spacings are too numerous for reliable correlation with the observed values.

Electron Diffraction Data

It is well established that sepiolite has a fibrous lath-like morphology and this has been confirmed in the present work. However, the laths are far from being equally well developed in all specimens. In the case of the Eski Chehir material, the individual fibers are very small and poorly developed, and the single crystal diffraction patterns are also very poor. Figure 2(b) shows a typical diffraction diagram for the Eski Chehir sepiolite.

The well developed and well crystallized fibers of sepiolite from Little

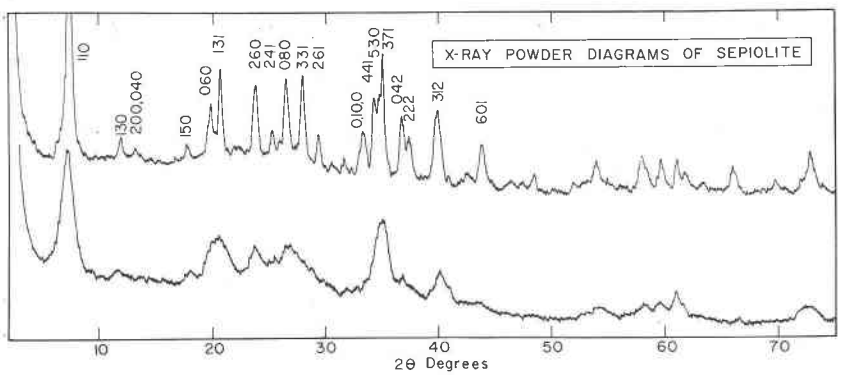

Fig. 1. X-ray diffractometer records of sepiolites from Little Cottonwood, Utah (upper curve), and Eski Chehir, Asia Minor (lower curve).

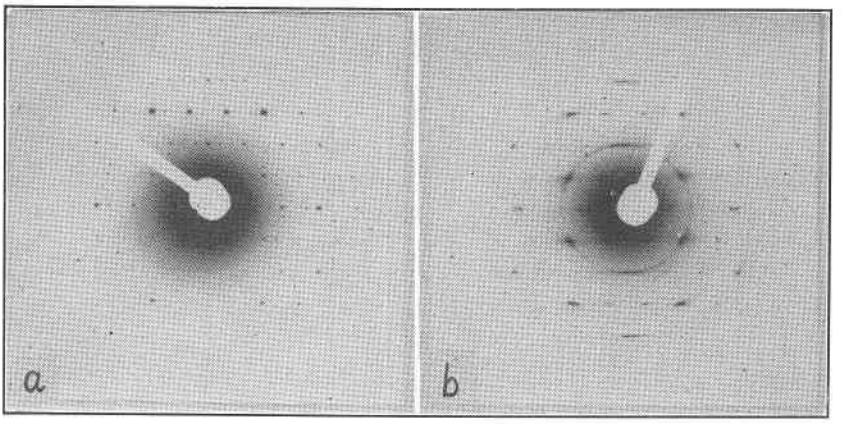

FIG. 2. Single crystal electron diffraction patterns of sepiolite from (*a*) Little Cottonwood, Utah, (*b*) Eski Chehir, Asia Minor.

Cottonwood, Utah, give excellent single crystal diagrams and a typical example is shown in Fig. 2(*a*). From enlargements of several of these patterns it is readily found that the diffraction spots lie on a rectangular net with sides about 1/5.3 Å and 1/26.8 Å to within $\pm 1\%$. Therefore the lath-shaped crystals lie with their *b* and *c* axes, i.e., the (100) plane, on the stage of the instrument. The usefulness of single-crystal electron diffraction data in establishing unit cell parameters when only powder or, at the best, fiber diagrams are obtainable with *x*-rays, is well illustrated by this example.

In terms of the structure described by Nagy and Bradley, it can now be said that the individual crystals develop primarily along the *c*-axis (fiber axis) and secondarily in the plane of the mica-like units composing the structural blocks. The laths are relatively thin in the direction of the *a*-axis, and the stacking of the mica-like units normal to the usual cleavage plane is the least favored growth process. Figure 3 illustrates sche-

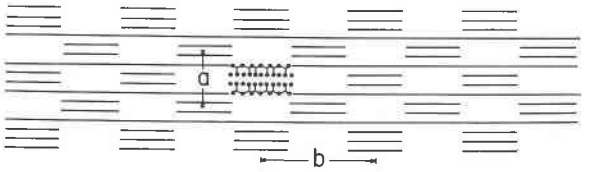

FIG. 3. Schematic representation of the cross-section of a sepiolite fiber, showing greater development in the *b* than in the *a* direction. The content of one unit cell is indicated schematically; the horizontal lines represent layers of oxygen atoms.

TABLE 1. OBSERVED AND CALCULATED *X*-RAY POWDER DATA FOR SEPIOLITE

hkl	*d*(Calc.) Å	*d*(Obs.) Å (1)	(2)	(3)	(4)
020	13.48	—	—	—	—
110	12.07	12.05 (100)	12.1 (100)	12.1 (100)	12.3 (60)
130	7.482	7.47 (10)	7.5 (7)	7.7 (5B)	7.6 (5)
200	6.750	6.73 (5)	6.7 (4B)	6.7 (5B)	—
040	6.742				
220	6.036	—	—	—	—
001	5.255	—	—	—	—
150	5.008	5.01 (7)	5.04 (3B)	5.0 (5B)	4.9 (6B)
021	4.896	—	—	—	—
111	4.818	—	—	—	—
240	4.780	—	—	—	—
060	4.495	4.498 (25)	4.49 (25)	4.47 (18)	4.5 (20NR)
310	4.438	—	—	—	
131	4.301	4.306 (40)	4.29 (35)	4.31 (25)	4.3
201	4.146	—	—	4.17 (5)	—
041	4.145	—	—	—	—
330	4.023	4.022 (7)	4.02 (7)	—	—
221	3.963	—	—	—	—
260	3.741	3.750 (30)	3.738 (25)	3.738 (20)	3.746 (20B)
170	3.705	—	—	—	—
151	3.626	—	—	—	—
241	3.532	3.533 (12)	3.506 (5)	—	3.49 (5)
350	3.455	—	—	—	—
061	3.416	—	—	—	—
311	3.391	—	—	—	—
400	3.375	—	—	—	—
080	3.370	3.366 (30)	3.339 (45)	3.339 (35)	3.34 (20NR,B)
420	3.274	—	—	—	
331	3.195	3.196 (35)	3.181 (15B)	3.187 (12)	
261	3.048	3.050 (12)	—	3.048 (5)	
171	3.028	—	—	—	
440	3.018	—	—	—	
280	3.016	—	—	—	
370	2.928	2.932 (4)	2.950 (5)	—	2.98
190	2.925	—	—	—	—
351	2.887	—	—	—	—
401	2.840	—	—	—	—
081	2.837	2.825 (7)	—	—	—
421	2.778	2.771 (4)	—	2.79? (4)	—
460	2.699	—	—	—	—
0.10.0	2.697	2.691 (20)	2.66 (8NR)	2.675 (8NR)	2.67 (2.56(40NR,B)
510	2.687				
002	2.627	—	—	—	
441	2.618	2.617 (30)	2.59 (45NR)	2.59 (40NR)	
281	2.617				
530	2.586	2.586 (NR)			
022	2.580	—			
112	2.567	2.560 (55)			
371	2.557				
191	2.556		2.56	2.56	
2.10.0	2.505	—	—	—	
390	2.495	—	—	—	
132	2.479	2.479 (5)	—	—	2.49

TABLE 1 (*continued*)

hkl	*d*(Calc.) Å	*d*(Obs.) Å (1)	(2)	(3)	(4)
202	2.448	2.449 (25)	2.43 (20NR)	2.44 (15NR)	2.43 (10NR)
042	2.448				
550	2.414				
1, 11, 0	2.412	2.406 (15)	2.395	2.39	2.36
222	2.409				
461	2.401				
062	2.268	2.263 (30)	2.256 (20)	2.259 (18B)	2.24 (20B)
312	2.261				
2.10.1	2.260				
620	2.220	2.206 (3)	—	—	—
570	2.211				
332	2.200				
640	2.134	2.125 (7)	2.117 (4B)	2.117 (5)	—
2, 12, 0	2.130				
4, 10, 0	2.107				
402	2.073				
082	2.072	2.069 (20)	2.060 (10)	2.071 (7B)	2.08 (6B)
601	2.069				
571	2.038	2.033 (4)	—	—	—
		1.957 (4)	—	—	—
		1.921 (2)	—	—	—
		1.881 (7)	1.873 (4)	—	—
		1.818 (2)	—	—	—
		1.760 (6)	—	—	—
		1.700 (10)	1.716 (7)	1.722 (5)	—
		—	1.691 (10)	1.692 (8)	1.69 (5B)
		1.637 (3)	—	—	—
		1.592 (10)	1.598 (4)	1.583 (9B)	1.58 (7)
		1.550 (15)	1.578 (7)	1.548 (10)	1.551 (10)
			1.540 (8)		
		1.518 (15)	1.517 (15)	1.517 (15)	1.517 (14)
		1.502 (8)	—	—	—
		1.468 (4)	1.465 (3)	—	—
		1.416 (9)	1.406 (4)	1.412 (4B)	—
		1.349 (6)	—	—	—
		1.312 (6)	—	1.310 (4B)	—
		1.299 (15)	—	1.296 (10)	—

Experimental data for sepiolites from:
(1) Little Cottonwood, Utah.
(2) Vallecas, Spain.
(3) Kenya.
(4) Eski Chehir, Asia Minor.
Intensities in ( ).
B signifies "broad"; NR "not resolved."

matically a cross-section of a sepiolite fiber with greater development in the *b* than in the *a*-direction. The length of the fiber normal to the diagram may be 10–100 times greater than the cross-section.

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Note added Oct. 1, 1958

At the time of writing the preceding article, the writer was unaware that Preisinger, in collaboration with Brauner, had already published a full account of the structure determination of sepiolite (K. Brauner and A. Preisinger, *Tschermaks min. u. pet. Mitte.*, **6**, 120–140, 1956). The abstract by Preisinger makes no reference to this publication.

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