

## Cavansite and Pentagonite, New Dimorphous Calcium Vanadium Silicate Minerals From Oregon

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### Abstract

Cavansite,  $\text{Ca}(\text{VO})(\text{Si}_4\text{O}_{10}) \cdot 4\text{H}_2\text{O}$ , a new mineral found in cavities and veinlets in basalt and vugs in tuff at Goble and at Owyhee Dam in Oregon, occurs as radiating greenish-blue prismatic crystals associated with calcite, analcime, thomsonite, heulandite, stilbite, and apophyllite. The crystals are orthorhombic, space group  $Pcmm$  ( $D_{2h}^{10}$ ), and have a unit cell with  $a = 9.778(3)$ ,  $b = 13.678(4)$ ,  $c = 9.601(2)$  Å, containing 4 formula units. They are optically biaxial positive and strongly pleochroic. The composition was determined by X-ray fluorescence analysis and crystal structure analysis. Pentagonite, a dimorph of cavansite, was found at Owyhee Dam in prismatic crystals twinned to form fivelings with star-shaped cross sections. It is also orthorhombic, space group  $Ccm2_1$  ( $C_{2v}^{12}$ ), and has a unit cell with  $a = 10.298(4)$ ,  $b = 13.999(7)$  and  $c = 8.891(2)$  Å, containing 4 formula units. These cell dimensions, and those for cavansite, are typical and tend to vary over a small range, presumably because of varying zeolitic water content. The crystals are optically very similar to cavansite, but are biaxial negative. Both cavansite and pentagonite have silicate layer structures in which the layers are held together by  $\text{VO}^{2+}$  groups and  $\text{Ca}^{2+}$  ions, but they differ in the way the  $\text{SiO}_4$  tetrahedra link to form the layers. The crystal chemical study fully accounts for the morphological and physical properties of these unusual minerals.

### Introduction

In the fall of 1960, Mr. and Mrs. Leslie Perrigo of Fruitland, Idaho, observed a blue mineral partly coating a large rock face in a new roadcut near Owyhee Dam, Malheur County, Oregon. Together with Mr. and Mrs. Frank Zimmerman of Payette, Idaho, they collected some of the material the following spring and sent it to Dr. Paul Desautels of the United States National Museum, who reported that it was probably a new mineral.

Mr. John Cowles of Rainier, Oregon, in February 1963, discovered a similar-appearing blue mineral in a quarry near Goble, Columbia County, Oregon, approximately 350 miles northwest of the Owyhee locality. He presented some of his material to one of the authors (L. W. Staples) for identification in the fall of that year, and it was determined to be a new mineral. Subsequently, some of the Owyhee material was obtained and comparison of specimens from both localities proved them to be the same mineral. Our study of the new mineral has included

a crystal structure analysis, which shows it to be a layer silicate of a completely novel type. The name, cavansite (ca'-van-site) has been selected as a mnemonic word from the chemical composition. The mineral and the name have been approved by the Commission on New Mineral Names, International Mineralogical Association.

During the crystallographic study, one small specimen from Owyhee Dam was examined which contained crystals that were clearly twinned. These proved to be different from cavansite, though they have very similar appearance and physical properties, and the same chemical composition. Crystal structure analysis showed the twinned crystals also have a silicate layer structure, but with a different type of layer than that of cavansite. The twinned material, therefore, is a dimorph of cavansite and constitutes yet another new mineral species. We have called it pentagonite (pen-ta'-gon-ite) in allusion to its striking twin aspect; this name has also been approved by the Commission on New Mineral Names.

In this paper we present a description of the mineralogy and paragenesis of these two new vanadium silicate minerals. The details of the crystal structures are given in a separate but accompanying paper (Evans, 1973).

### Occurrence

Cavansite and pentagonite are found in a brown tuff in a roadcut 2.1 miles by road south of Owyhee Dam and about a quarter mile north of Gordon Gulch, on the east side of the reservoir. The locality lies within the boundaries of the Lake Owyhee State Park. The mineral partially fills a fault fissure which strikes N. 40° E. and is almost vertical. Calcite filling of the fault shows slickensides, but rosettes of cavansite on the calcite have not been deformed. The tuff is in the Sucker Creek Formation (Corcoran *et al*, 1962; Kittleman *et al*, 1965) of late Miocene age and is overlain by the Owyhee Basalt, feeders of which frequently cut the Sucker Creek Formation (Corcoran, 1965). On the east side of the highway for a distance of about 90 feet, the roadcut exposes a face 17 feet high with a coating 2–3 mm thick of cavansite, pentagonite, and associated minerals. Veinlets of cavansite occur in the tuff for another 300 feet to the south, but here the mineral is poorly exposed due to lack of excavation.

At Goble, cavansite is found in the Charles W. Chapman quarry, 0.5 miles west of U. S. Highway 30 on Neer Road. The quarry is in a brown to gray vesicular basalt and red tuff breccia belonging to the Goble Volcanic Series of late Eocene age (Wilkinson *et al*, 1946). The cavansite occurs as cavity fillings, in amygdules, and in calcite veinlets. Other quarries in the area have blue stains on some of the secondary minerals, but only in the Chapman quarry was cavansite found.

Pentagonite, found so far only at Owyhee Dam, seems to have formed at a later stage than cavansite, but their mineral associations and mode of occurrence are nearly identical.

A few small specimens of cavansite and pentagonite from Owyhee Dam, Oregon, and a smaller amount of cavansite from Goble, have been placed in the collections of the Smithsonian Institution at the U. S. National Museum, Washington, D. C.

### Paragenesis

At Owyhee Dam the principal associates of cavansite and pentagonite are calcite, heulandite, stilbite, analcime, and apophyllite. The apophyllite occurs

as very small (*ca* 0.3 mm) crystals, frequently doubly terminated, and coating the heulandite. The calcite is of two generations, an amber-colored variety in rhombohedrons which preceded the cavansite and a colorless variety which is much later. Both heulandite and analcime enclose pentagonite and cavansite and as a result assume a deep blue color, giving the impression that more cavansite is present than is actually the case. The pentagonite is probably of two generations. Gmelinite is found lining cavities at a location about 500 feet above the cavansite occurrence. Its paragenetic relation to the other minerals is not known. The sequence of deposition at Owyhee Dam appears to be: (1) amber calcite, (2) cavansite, (3) pentagonite and heulandite, (4) stilbite, (5) pentagonite, (6) calcite, (7) analcime, (8) apophyllite.

One specimen was found that has both cavansite and pentagonite together. Here the cavansite is perched in bold radiating groups on calcite, over which large, clear, colorless crystals of heulandite have formed. Smaller, long prismatic crystals of pentagonite fill the open space above the calcite and cavansite, and penetrate the heulandite crystals in all directions. This association of minerals suggests that this specimen during its formation passed through the temperature of transition between cavansite and pentagonite, and that the latter is the low-temperature form.

At Goble, the sequence of deposition is more difficult to determine but a common sequence is (1) calcite, (2) heulandite, (3) thomsonite, (4) cavansite, (5) calcite. Native copper is also present in some of the specimens and the late calcite may enclose both cavansite and copper. Because the cavansite was deposited about the same time and presumably under the same conditions as calcite and the zeolites, it is difficult to separate these minerals in order to obtain a pure sample for analysis.

The source of the vanadium for these vanadium minerals is not known. At Owyhee Reservoir, a rhyolite intrudes and overlies Kittleman's Sucker Creek Formation and basalt feeder dikes of the Owyhee Basalt prominently cut through the Sucker Creek tuff. The introduction of vanadium may be related to these intrusions.

### Chemical Compositions

Because of the intimate association of cavansite with other minerals, especially zeolites, it was difficult to obtain a sample large enough for analysis by

wet methods. The Oregon State Department of Geology and Mineral Industries kindly supplied us with some analytical information obtained on a gross sample from Goble. Their spectrographic test showed Si, V, and Ca as major constituents. Their wet analysis did not conform well stoichiometrically to any reasonable formula. Since the purity of the large sample used was open to question, other means of analysis were sought.

X-ray fluorescence analysis carried out on a 20 mg sample proved to be more reliable. The heavy absorber-fusion technique (Rose *et al*, 1963) was used to prepare the sample and standards. The standards were prepared by mechanically incorporating  $V_2O_5$  with a set of calcium silicate reference materials to give the appropriate concentration ranges of CaO (9.8 to 29.4%),  $V_2O_5$  (7.5 to 22.5%), and  $SiO_2$  (25 to 75%). The analysis was carried out with a vacuum spectrometer (built by the General Electric Company) equipped with a chromium target X-ray tube (operated at 40 ma and 60 kv), an ethylene diamine tartrate analyzing crystal, and a flow proportional counter.

The elements Ca, V, and Si were determined using the  $K\alpha$  line of each and counting for a period long enough to give approximately 10,000 counts on the lowest standard. In this way the following composition was found:

	Wt %	Mole ratios
CaO	11.5	.998
VO <sub>2</sub>	17.1	1.002
SiO <sub>2</sub>	49.4	4.000
H <sub>2</sub> O	(21.0)	
Rem.	.8	
Total	99.8	

The water content was estimated by the Oregon State Department of Geology and Mineral Industries Laboratory (see above). The mole ratios clearly establish a formula of the form  $CaO \cdot VO_2 \cdot 4SiO_2 \cdot nH_2O$ , but the water content was relatively uncertain and the value of  $n$  could not be determined by the usual analytical methods. The amount found above suggests  $n = 6$ , but this predicts a specific gravity of 2.52 from the X-ray unit cell, which compares very poorly with the highest measured value of 2.31 (see below). The true ideal water content was finally established by crystal structure analysis, giving the formula for cavansite:  $CaO \cdot VO_2 \cdot 4SiO_2 \cdot 4H_2O$ , or  $Ca(VO)(Si_4O_{10}) \cdot 4H_2O$ .

The composition of the twinned material, pen-

tagonite, was estimated by an alternate X-ray fluorescence technique, using a General Electric Company Model XRD-6 Semifocussing Spectrometer. In it the specimen, which may be less than 1 mm in diameter, is bathed in intense tungsten (or chromium) radiation from a type EA-75 dual target X-ray tube, and the excited radiation is collimated by a 1 mm beam tunnel. The collimated X-radiation is then analyzed with a curved LiF crystal and detected with a flow proportional counter. Four different analyzing crystals were available to provide optimum focussing over a wide range of wave lengths. The apparatus and procedure have been described by Rose *et al* (1969) and its adaptation for study of small single crystals used in X-ray crystallography by Evans and Christian (1972). This technique is not capable of the accuracy of that described above for cavansite, but can yield (with a sample of  $\sim 0.1 \mu g$ ) semi-quantitative information that can decisively determine critical ratios. In this case, a single crystal of pentagonite was compared with a similar crystal of cavansite, and one of pascoite ( $Ca_3V_{10}O_{28} \cdot 17H_2O$ ). The relative peak heights for Ca, V, and Si for the different crystals indicated clearly that the ratio of Ca:V:Si is the same for both cavansite and pentagonite.

The amount of water present was again the unknown quantity. The question was answered in this case by comparison of the unit-cell volumes of cavansite and pentagonite (Table 1). Since they are nearly identical ( $\Delta V$  for one  $H_2O$  unit would be  $\sim 80 \text{ \AA}^3$ ), we may safely conclude that the chemical formulas of the two species are the same.

The final conclusive confirmation of the formulation  $CaO \cdot VO_2 \cdot 4SiO_2 \cdot 4H_2O$  for cavansite and pentagonite was provided by complete crystal structure analyses, described in the paper (Evans, 1973) that follows. In all these considerations the oxidation state of vanadium was assumed to be IV, mainly because of the weak red optical absorption shown by both materials, which is characteristic of the  $VO^{2+}$  cation in many of its compounds. Again, this assumption was confirmed by the crystal structure determination.

For chemical tests, the following properties may be noted. Cavansite and pentagonite are difficultly soluble in acids. In  $HNO_3$ , the blue color of the minerals bleaches out and turns olive brown. A fine white crystalline precipitate is slowly formed on a microscope slide when the minerals are heated in dilute  $H_2SO_4$ .

TABLE 1. Crystal Data for Cavansite and Pentagonite

	Cavansite	Pentagonite
Crystal system	orthorhombic	orthorhombic
Space group	$Fmm$ ( $D_{2v}^{16}$ )	$Cm2_1$ ( $C_{2v}^{12}$ )
Axial ratios (from X-ray powder data)		
$a:b:c$	0.7149:1:0.7019	0.7356:1:0.6351
Structure cell (from X-ray powder data)		
$a, \text{\AA}$	9.778±0.003	10.298±0.004
$b, \text{\AA}$	13.678±0.004	13.999±0.007
$c, \text{\AA}$	9.601±0.002	8.891±0.002
$V, \text{\AA}^3$	1284.1 ± 0.5	1281.8 ± 0.6
$Z$ , formula units	4	4
Specific gravity		
Calc (from X-ray)	2.33	2.33
Obs (Berman balance)	2.21-2.31	
Optics		
Character	Biax. positive	Biax. negative
Optic plane	$\perp a$	$\perp a$
Acute bisectrix	$\parallel c$	$\parallel b$
$X$	$\parallel b$ , colorless	$\parallel b$ , colorless
$Y$	$\parallel a$ , blue	$\parallel a$ , blue
$Z$	$\parallel c$ , colorless	$\parallel c$ , colorless
$n_X$	1.542±0.002	1.533±0.002
$n_Y$	1.544±0.002	1.544±0.002
$n_Z$	1.551±0.002	1.547±0.002
$2V$	$52 \pm 2^\circ$	$50 \pm 2^\circ$
Dispersion	$n < \nu$ , extreme	$n > \nu$ , strong

### Physical and Optical Properties

Cavansite forms single prismatic crystals usually ~0.05 mm in diameter and 0.5-1.0 mm in length. It also commonly forms spherulitic rosettes up to 5 mm in diameter. The color is a beautiful "brilliant greenish blue" (NBS color standard no. 168; Kelly and Judd, 1955). The luster is vitreous. Pentagonite is similar in habit, and is perhaps slightly less greenish in color.

The hardness has not been accurately determined because of the small size of the crystals, their brittleness, and their good cleavage. It appears to be about 3-4 on the Mohs scale. The cleavage is good parallel to  $b$  {010} for both cavansite and pentagonite.

The observed specific gravity for cavansite (Table 1) is the average of measurements made with the Berman balance on five separate samples. Considerable difficulty was encountered in getting fragments completely free of admixed zeolites. The specific gravity of pentagonite was not determined.

Cavansite and pentagonite have similar, though distinctly different, optical properties. Both are transparent, pleochroic blue, biaxial crystals elongated parallel to the  $c$  axis, which corresponds to the  $Z$  vibration direction. The indices of refraction are such that the acute bisectrix shifts from  $c$  in cavansite to  $b$  in pentagonite, and the strong dispersion is therefore reversed. The critical data (Table 1) were measured with the aid of a spindle stage (using white and sodium light) according to the method of Wilcox (1959). The cyclic twinning of pentagonite often has the appearance of polysynthetic twinning in polarized light, producing lamellae parallel to the elongation direction of the crystals.

### Crystal Morphology

Both cavansite and pentagonite are orthorhombic with unit cells that are somewhat similar, but distinctly different. The habit of both minerals is prismatic, elongated parallel to  $c$ , with dome terminations. As illustrated in Figure 1, the dominant forms for cavansite are  $m$  {110} and  $d$  {101}, and for pentagonite  $m$  {110},  $u$  {201}, and  $c$  {001}. Minor forms that have been observed for cavansite are  $b$  {010},  $c$  {001},  $a$  {100}, and  $p$  {111}; and for pentagonite,  $c$  {001} and  $a$  {100}. The axial ratios as determined from the X-ray study are given in Table 1. The characteristic inclination angle  $\rho$  for  $d$  on cavansite crystals is  $44.5^\circ$ , and for  $u$  on pentagonite crystals it is  $59.9^\circ$ .

Pentagonite is nearly always twinned by reflection across the prism  $m$  (110), which is the composition plane. The prism angle  $m \wedge m' = 72.7^\circ$  so that multiple twinning leads to groups which closely simulate five-fold symmetry. This very characteristic habit is

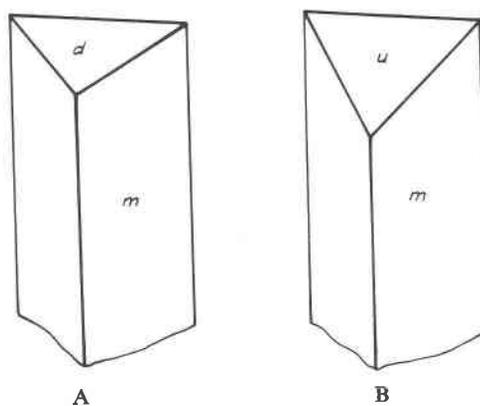


FIG. 1. Single crystal habit of (A) cavansite, and (B) pentagonite. Forms are:  $m$  {110},  $d$  {101},  $u$  {201}.

clearly shown in Figure 2. Two terminal habits are apparent, one showing chisel-shaped ends formed by the steep dome  $u$ , and the other showing only the blunt pedion. From the structure analysis, the symmetry is known to be polar ( $mm2$ ), and these two habits probably represent opposite ends of the same crystal type (no doubly terminated crystals have been found). Figure 3 illustrates these two aspects, and shows the pedion  $c$  on both, but when associated with the dome (Fig. 3A) this form is generally rough and gives no optical signal. In the twins, the dome is very erratic in its development.

Although the prism angle  $m \wedge m'$  for cavansite is  $71.1^\circ$  and thus close to the  $72.7^\circ$  angle observed for pentagonite, no twinning has yet been observed in this mineral.

### X-ray Crystallography

The unit cells and symmetries of cavansite and pentagonite were determined from Buerger precession photographs of the appropriate reciprocal net planes. The systematic absences of reflections in cavansite indicated the space groups  $Pcmn$  or  $Pc2_1n$ , and in pentagonite the space groups  $Ccmm$ ,  $Ccm2_1$ , or  $Cc2m$ . The structure analyses have shown that the centrosymmetric group is appropriate for cavansite, but that a noncentrosymmetric group is required for pentagonite. The correct space groups so determined are given in Table 1.

Refinement of the unit-cell dimensions, measured

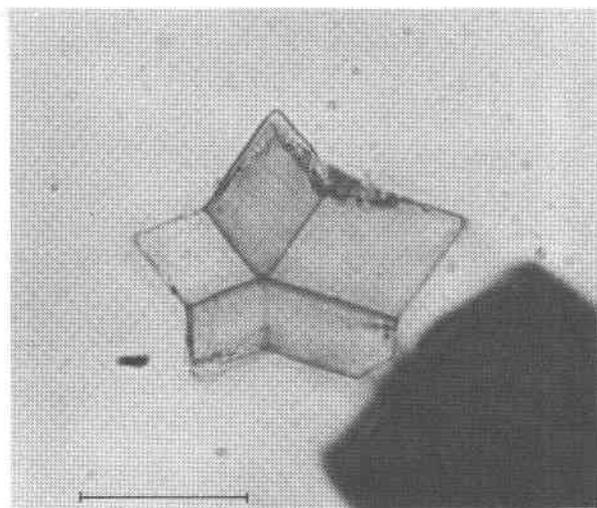


FIG. 2. Photomicrograph of pentagonite fiveling immersed in oil ( $n = 1.532$ ), showing end view of prismatic crystal. The pleochroism is apparent (plane of polarized light is vertical). The bar represents 0.05 mm.

initially on the Buerger precession photographs, was carried out on a set of  $2\theta$  values obtained from focussed X-ray powder diffraction patterns made with a Hägg-Guinier (xdc-700) camera using strictly monochromatized  $CrK\alpha_1$  radiation. The least-squares calculation (Evans *et al.*, 1963) was based on 35 indexed lines for cavansite and 19 lines for pentagonite. The resulting unit-cell constants are given in Table 1 and the X-ray powder data in Table 2. The powder data indexing was confirmed for both minerals by the single-crystal intensity data, which were determined for the three-dimensional structure analyses. Complete correspondence was found, and ambiguities of indexing were largely eliminated. Therefore, only observed powder reflections (with the corresponding calculated  $d$  spacings) are listed in Table 2.

Measurements on single crystals give unit cell parameters that differ slightly from those obtained from powder data (Table 1). For example, measurements on the Picker diffractometer with the single crystals used for structure determination (Evans,

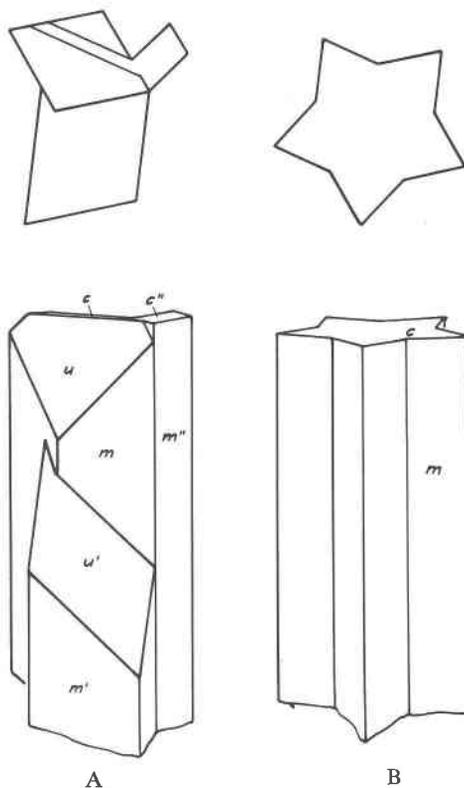


FIG. 3. Multiple twinned crystal habits of pentagonite. Two types shown are typical bladed habit (A), and idealized blunt habit (B).

TABLE 2. X-ray Powder Data for Cavansite and Pentagonite from Owyhee Dam, Oregon

Cavansite				Pentagonite			
hkl	d(calc)	d(obs)	I	hkl	d(calc)	d(obs)	I
110	7.954	7.964	100	110	8.295	8.298	70
101	6.851	6.854	50	020	7.000	7.006	2
111	6.125	6.132	25	111	6.065	6.071	100
121	4.840	4.841	6	201	4.456	4.446	25
021	4.530	4.531	13	002	4.446		
201	4.357	4.362	6	112	3.918	3.920	100
102	4.309	4.310	6	221	3.759	3.755	100
211	4.151	4.158	6	022	3.753		
220	3.977	3.978	5	040	3.500	3.500	36
022	3.930	3.930	25	202	3.365	3.364	9
122	3.646	3.647	3	132	3.072	3.072	2
040	3.419	3.420	25	222	3.033	3.034	9
212	3.323	3.323	2	330	2.765	2.764	13
310	3.170	3.171	3	241	2.752	2.752	9
132	3.132	3.133	3	042	2.750		
301	3.086	3.087	5	331	2.640	2.640	25
222	3.063	3.062	13	203	2.569	2.569	36
141	3.059						
103	3.042	3.040	2	401	2.473	2.475	2
113	2.969	2.970	1	133	2.431	2.431	25
321	2.813	2.813	3	242	2.426	2.425	7
123	2.779	2.779	25	332	2.348	2.348	4
232	2.739	2.739	5	004	2.223	2.223	9
302	2.697	2.696	6	313	2.215	2.213	2
312	2.646	2.648	2	044	1.876	1.876	9
150	2.634	2.635	2				
223	2.493	2.495	2				
400	2.444	2.444	3				
004	2.400	2.402	2				
052	2.377	2.375	2				
014	2.364	2.364	9				
332	2.321	2.320	3				
233	2.309	2.309	9				
114	2.298	2.298	2				
143	2.273	2.274	5				
034	2.124	2.123	2				
243	2.108	2.109	3				
350	2.095	2.095	3				
440	1.9886	1.9883	1				
403	1.9426	1.9421	1				
144	1.9261	1.9265	2				
170	1.9161	1.9150	1				
511	1.8977	1.8975	3				
105	1.8843	1.8838	2				
163	1.8242	1.8237	2				

## Notes:

1. d(calc) in Å are from unit cell parameters given in Table 1.
2. d(obs) in Å are from Hagg-Guinier patterns made with CrK $\alpha$ , rad.,  $\lambda = 2.28962$  Å.

1973) gave for cavansite  $a=9.792$ ,  $b=13.644$ ,  $c=9.629$ Å and for pentagonite  $a=10.386$ ,  $b=14.046$ ,  $c=8.975$ Å. These variations probably arise from zeolitic variation in the amount of water in the structure, which is accompanied by a change in the dimensions of the unit cell, especially along the  $c$  axis. The variation of several specific gravity measurements of cavansite over a range below the value predicted by the unit cell dimensions (Table 1) is also probably related to the zeolitic nature of the mineral.

### Crystal Chemistry

Although the general chemistry, physical properties, and mineralogy of cavansite were known at

an early stage, the nature of the mineral was not really understood until its crystal structure had been revealed. From the formula we could expect that a silicate layer structure is present, unless this was one of the unusual cases where the SiO $_4$  group incorporates an OH radical. The structure analysis proved that the structure is indeed a layer type, and that all of the hydrogen is carried by rather loosely bound, interlayer water molecules. The structure study also established conclusively that ideally there are 4H $_2$ O per formula unit.

The structure analysis described in the accompanying paper (Evans, 1973) reveals the dimorphism of cavansite and pentagonite to be based on the difference in linkages in the silicate layer. Zig-zag (SiO $_3$ ) $_n$  chains are discernible in both, but in cavansite these chains are joined laterally into sheets made up of 4-fold and 8-fold rings, while in pentagonite they are differently joined so that only 6-fold rings

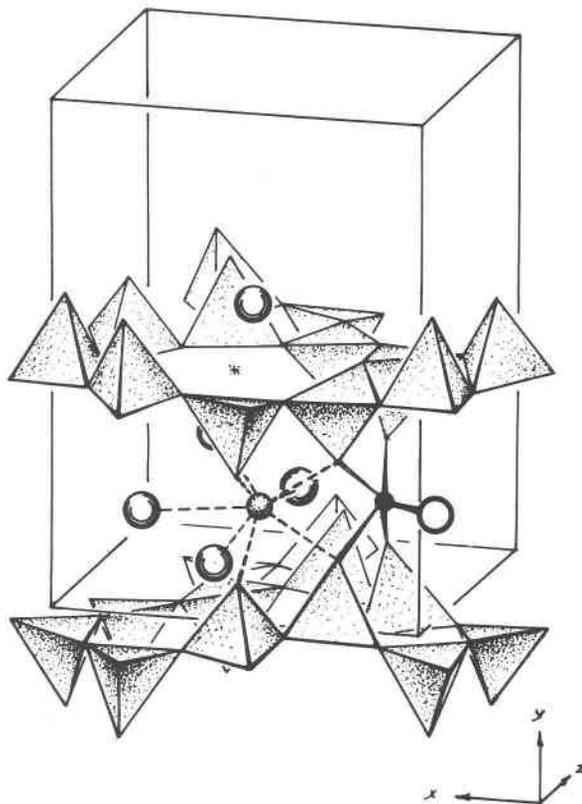


FIG. 4. Pictorial view of the crystal structure of cavansite with the silicate layers in horizontal position ( $b$  axis vertical). The tetrahedra represent SiO $_4$  groups, the filled circle with attached open circle is a VO $_2^+$  group, the stippled circle is a Ca $^{2+}$  ion, and the line-shaded circles are H $_2$ O molecules.

are formed. In both structures the unlinked tetrahedral apices of the chains are coordinated in pairs by  $\text{VO}^{2+}$  groups, which tie the layers together. Thus, the vanadium atoms achieve their characteristic five-fold, square-pyramidal coordination. Large cavities in the structure are filled zeolitically by  $\text{Ca}^{2+}$  ions and  $\text{H}_2\text{O}$  molecules. These structural features are pictorially illustrated for cavansite in Figure 4.

All of the chemical and physical properties of the two minerals are satisfactorily explained by their crystal structures. In particular, the special geometry of the pentagonite crystal structure readily permits twinning at an angle near  $72^\circ$ , thus giving rise to the striking fivelings commonly observed. The structure study (Evans, 1973) has shown that no such twinning mechanism can be imagined for the cavansite structure, and in fact, no twins of this mineral have as yet been observed.

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