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HYDROTHERMAL SYNTHESIS OF VEATCHITE AND ITS CALCIUM ISOMORPH,

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ABSTRACT

The hydrated strontium borate, veatchite ($SrO \cdot 3B_2O_3 \cdot 2H_2O$), and its calcium isomorph ($CaO \cdot 3B_2O_3 \cdot 2H_2O$) have been prepared by hydrothermal synthesis at 260 °C. Veatchite was prepared by the reaction of $Sr(OH)_2$ and H_3BO_3 solution and also by the conversion of tunellite ($SrO \cdot 3B_2O_3 \cdot 4H_2O$) in H_3BO_3 solution at 260 °C. The calcium isomorph was prepared in a similar manner by the reaction of $Ca(OH)_2$ and H_3BO_3 solution and also by the conversion of gowerite ($CaO \cdot 3B_2O_3 \cdot 5H_2O$) in H_3BO_3 solution at 260 °C. The observed densities of the synthetic veatchite and the calcium isomorph, as determined with a pycnometer and using water as the liquid, were 2.77 ± 0.04 and 2.31 ± 0.02 g/[illegible] cc respectively, for 0.5-g samples of fine crystals. The highest and lowest indices of refraction (white light) for the synthetic veatchite and its calcium isomorph were determined to be 1.546 and 1.619 ± 0.003, and 1.555 and 1.620 ± 0.002, respectively.

FOREWORD

The Naval Ordnance Laboratory Corona is making a continuing study of the chemistry and synthesis of crystals of ferroelectric colemanite ($2CaO \cdot 3B_2O_3 \cdot 5H_2O$), its strontium isomorph ($2SrO \cdot 3B_2O_3 \cdot 5H_2O$), and other related calcium and strontium borate hydrates of potential use in ferroelectric devices. As part of this study, the chemistry and methods of synthesis of veatchite ($SrO \cdot 3B_2O_3 \cdot 2H_2O$) and its calcium isomorph ($CaO \cdot 3B_2O_3 \cdot 2H_2O$) are reported herein. The dielectric, ferroelectric, and pyroelectric properties of the materials synthesized will be reported later. All the above work is authorized by ONR Contract PO 1-0008 NR 048-119.

C. J. HUMPHREYS
Head, Research Department

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INTRODUCTION

The synthesis of colemanite single crystals in 1959 and the preparation of deuterated colemanite in 1961 have been reported in NOLC Technical Memorandums (Refs. 1, 2, 3), and in 1962 a comparison of the ferroelectric and pyroelectric properties of mineral and synthetic colemanite was made (Ref. 4). The synthesis of the hydrated calcium borates was then investigated further to improve the quality and size of the crystals produced, and a limited study was made of the hydrothermal reactions that produce hydrated calcium and strontium borates and the transformation these borates undergo with increased temperature.

The work reported in this document is only a small part of the overall investigation of the calcium and strontium borate hydrates that might prove of interest to those people who are concerned with ferroelectric devices. Since, to the author's knowledge, there has been no previous laboratory synthesis of the mineral veatchite ($SrO \cdot 3B_2O_3 \cdot 2H_2O$), and no mention of the existence of a calcium isomorph ($CaO \cdot 3B_2O_3 \cdot 2H_2O$), this work is considered sufficiently important to be reported separately from the results of the entire investigation, which will appear in a report now in process of publication (Ref. 5).

PREPARATION OF SYNTHETIC VEATCHITE

It was found that synthetic veatchite could be prepared by two methods. By the first method, strontium hydroxide is reacted with boric acid in aqueous solution at 260°C for 64 hours. By the second method, the veatchite is prepared by the transformation of synthetic "tunellite" ($SrO \cdot 3B_2O_3 \cdot 4H_2O$) in boric acid solution at 260°C for 40 hours. The second method produces the best quality of crystals.

Method I. Reaction of $Sr(OH)_2$ and H_3BO_3. In this method and also in Method II, the reaction is carried out in a sealed, stainless steel bomb, which has a capacity of 76 ml and is equipped with a platinum liner. The platinum liner is not absolutely necessary, but it serves to reduce the contamination of the product. The reaction mixture consists of 8.50 ml H_2O (CO_2 free), 1.42 g anhydrous SrO, and 7.30 g boric acid crystals. The reaction is completed within 64 hours at 260°C to form veatchite.

Method II. Transformation of tunellite ($SrO \cdot 3B_2O_3 \cdot 4H_2O$) in boric acid solution. The reaction mixture consists of 22.6 ml H_2O (CO_2 free), 1.89 g synthetic tunellite, and 4.52 g boric acid crystals. The reaction is completed within 40 hours at 260°C to form veatchite. In the absence of boric acid, this reaction does not produce veatchite.

Table 1 gives the X-ray diffraction analysis for both the synthetic veatchite produced by the above methods and the calcium isomorph of veatchite discussed below. For comparison purposes, X-ray data are presented from investigations of the mineral veatchite by others (Refs. 6, 7, 8).

PREPARATION OF THE CALCIUM ISOMORPH OF VEATCHITE

Reactions similar to those described above produce the calcium isomorph ($CaO \cdot 3B_2O_3 \cdot 2H_2O$). The reactions are identical, except that for Method I an equivalent amount of $Ca(OH)_2$ is substituted for the SrO, and in Method II gowerite ($CaO \cdot 3B_2O_3 \cdot 5H_2O$) is substituted for tunellite. The reaction time is reduced to 40 hours at 260°C in the formation of the calcium compound, since longer reaction time favors the formation of other reaction products. See Table 1 for the X-ray diffraction data for the synthetic calcium isomorph produced by these reactions. Details of other reaction times and resultant products will be published in the forthcoming report on the entire investigation (Ref. 5).

This isomorph can also be produced by the conversion reaction of synthetic ginorite ($2CaO \cdot 7B_2O_3 \cdot 8H_2O$) in boric acid solution in a manner identical with that given for the transformation of gowerite. In the presence of boric acid solution of the strength used in the above reactions, gowerite is completely transformed to ginorite at 170°C and to the veatchite isomorph at 260°C.

The compound $CaO \cdot 3B_2O_3 \cdot 2H_2O$ is also one component of the reaction product resulting when a mixture containing 0.20 g colemanite and 1.00 g priceite is reacted for 5 days at 200°C with 50 g of 20% boric acid solution in a 76-ml bomb. The second component of this reaction product will be discussed in the report cited in Ref. 5.

DENSITY AND REFRACTIVE INDEX OF SYNTHETIC VEATCHITE AND THE CALCIUM ISOMORPH

Two determinations of density were made with a pycnometer on separate samples of the synthetic veatchite crystals, using water as the liquid. The values obtained for the two specimens (approximately 0.5 g each) of fine crystals were 2.73 and 2.80 g/cm^3. This compares favorably with the value of 2.78 ± 0.03 reported by Clark, Mrose, Perloff, and Burley (Ref. 6).

Two determinations of density of the synthetic $CaO \cdot 3B_2O_3 \cdot 2H_2O$ were also made by the pycnometer method. The values obtained for the two specimens (approximately 0.5 g each) of fine crystals were 2.29 and 2.32 g/cm^3.

The highest and lowest refractive indices were determined for the synthetic veatchite and $CaO \cdot 3B_2O_3 \cdot 2H_2O$ by using the immersion method and white light. The values for synthetic veatchite are 1.546 and 1.619 (± 0.003), and for the calcium isomorph, 1.555 and 1.620 (± 0.002).

TABLE 1. X-Ray Powder Data for Synthetic Veatchite, the Calcium Isomorph of Veatchite, and Mineral Veatchite

Synthetic Veatchite— Parkerson, 1963		Calcium Isomorph of Veatchite— Parkerson, 1963		Mineral Veatchite							
				Stewart, Chalmers, and Phillips, 1954		Kramer and Allen, 1956		Clark, Mrose, Perloff, Burley, 1959			
(measured)		(measured)		(measured)		(measured)		(measured)		(calculated)	
I	d_{hkl}	I	d_{hkl}	I*	d_{hkl}	I	d_{hkl}	I	d_{hkl}	d_{hkl}	hkl
100	10.5	100	10.47	vs[1]	10.3	10	10.53	100	10.5	10.40	200
										5.870	020
										5.775	011
5	5.68	1	5.57	m	5.65	1	5.63	6	5.64	5.649	120
										5.609	$\bar{1}11$
1	5.43	1	5.27							5.521	111
										5.199	400
4	5.15	4	5.15	m	5.13	3	5.22	6	5.12	⎧5.115	$\bar{2}11$
		1	4.92							⎩5.112	220
1	4.79	1	4.79							4.985	211
1	4.52	1	4.48					2	4.51	⎧4.505	$\bar{3}11$
1	4.46	1	4.37	w	4.47					⎩4.480	320
1	4.13	1	4.11			1	4.20	2	4.37	4.372	311
2	3.88	>1	4.02			1	3.93	4	3.92	⎧3.924	$\bar{4}11$
2	3.83	>1	3.77	m	3.88					⎩3.892	420
1	3.67	1	3.69			1	3.69	3	3.81	3.807	411
22	3.48	18	3.42	w	3.48	10	3.47	20	3.47	3.466	600
										3.425	$\bar{5}11$

(Contd.)

NOTE: Footnotes are at end of table.

TABLE 1. (contd.)

Synthetic Veatchite—Parkerson, 1963		Calcium Isomorph of Veatchite—Parkerson, 1963		Mineral Veatchite							
				Stewart, Chalmers, and Phillips, 1954		Kramer and Allen, 1956		Clark, Mrose, Perloff, Burley, 1959			
(measured)		(measured)		(measured)		(measured)		(measured)		(calculated)	
I	d_{hkl}	I	d_{hkl}	I*	d_{hkl}	I	d_{hkl}	I	d_{hkl}	d_{hkl}	hkl
										3.394	520
9	3.37							3	3.37	3.370	031
										3.336	$\bar{1}31$
	3.33	14	3.28	vs^2	3.33	2	3.32	35	3.32	3.328	511
										3.318	131
										3.316	002
1	3.23	>1	3.22	w	3.20			3	3.22	3.223	$\bar{2}31$
										3.192	$\bar{2}02$
>1	3.15	>1	3.14							3.190	231
		3	3.09							3.128	202
		3	3.05							3.053	$\bar{3}31$
1	3.02									3.013	611
		>1	2.995	wb	2.99			3_b	3.00	3.010	331
1	2.986									2.985	620
		>1	2.947							2.935	040
1	2.938							2	2.936	2.933	611
										2.906	140
		3	2.882							2.887	022
8	2.864			m	2.87	3	2.88	9	2.865	2.872	$\bar{1}22$
										2.851	$\bar{4}31$

(Contd.)

NOTE: Footnotes are at end of table.

TABLE 1. (contd.)

Synthetic Veatchite— Parkerson, 1963		Calcium Isomorph of Veatchite— Parkerson, 1963		Mineral Veatchite						
				Stewart, Chalmers, and Phillips, 1954		Kramer and Allen, 1956		Clark, Mrose, Perloff, Burley, 1959			
(measured)		(measured)		(measured)		(measured)		(measured)		(calculated)	
I	d_{hkl}	I	d_{hkl}	I*	d_{hkl}	I	d_{hkl}	I	d_{hkl}	d_{hkl}	hkl
		2	2.846							2.848	122
										2.841	$\bar{4}02$
2	2.820	5	2.812							2.825	240
2	2.803							1	2.798	{2.806	431
										2.804	$\bar{2}22$
2	2.778			vvw	2.77		2.76	1	2.763	2.761	222
										2.753	402
>1	2.704			vvw	2.70		2.67	1	2.704	2.703	340
										2.695	$\bar{3}22$
>1	2.673									2.675	$\bar{7}11$
		1	2.642							2.651	720
										2.642	$\bar{5}31$
										2.637	322
										2.610	711
25	2.605	24	2.569	s^3	2.60	10	2.61	25	2.600	2.600	800
										2.596	531
		3	2.526					1	2.564	2.558	$\bar{4}22$
				vw	2.53					2.556	440
1	2.486	1	2.486					1	2.495	2.492	422
1	2.434	1	2.453							2.439	$\bar{6}02$

(Contd.)

NOTE: Footnotes are at end of table.

TABLE 1. (contd.)

Synthetic Veatchite—Parkerson, 1963		Calcium Isomorph of Veatchite—Parkerson, 1963		Mineral Veatchite							
				Stewart, Chalmers, and Phillips, 1954		Kramer and Allen, 1956		Clark, Mrose, Perloff, Burley, 1959			
(measured)		(measured)		(measured)		(measured)		(measured)		(calculated)	
I	d_{hkl}	I	d_{hkl}	I*	d_{hkl}	I	d_{hkl}	I	d_{hkl}	d_{hkl}	hkl
										2.438	$\overline{6}31$
										2.406	$\overline{5}22$
3	2.399			m	2.39	1	2.40	6	2.398	2.398	540, $\overline{8}11$
										2.395	631
1	2.372									2.377	820
										2.356	602
										2.344	811
		1	2.274							2.339	522
1	2.252	1	2.252					2	2.245		
5	2.204	1	2.210	m	2.20	1	2.21	3	2.204		
4	2.164	>1	2.174			1	2.17	2	2.171		
4	2.154	3	2.154	w	2.151	1	2.16	2	2.155		
2	2.130	1	2.125					2	2.115		
2	2.120			m	2.082	9	2.08	6	2.079		
8	2.079			m	2.037			4	2.045		
5	2.043	4	2.047	vw	1.996			4	2.029		
5	2.030	2	2.021					3b**	1.958		
4	2.009	3	1.996	m	1.936	1	1.924	3	1.925		
1	1.980							2	1.876		
1	1.959	2	1.967	vw	1.863			2	1.854		

(Contd.)

NOTE: Footnotes are at end of table.

TABLE 1. (contd.)

Synthetic Veatchite—Parkerson, 1963		Calcium Isomorph of Veatchite—Parkerson, 1963		Mineral Veatchite							
				Stewart, Chalmers, and Phillips, 1954		Kramer and Allen, 1956		Clark, Mrose, Perloff, Burley, 1959			
(measured)		(measured)		(measured)		(measured)		(measured)		(calculated)	
I	d_{hkl}	I	d_{hkl}	I*	d_{hkl}	I	d_{hkl}	I	d_{hkl}	d_{hkl}	hkl
2	1.943			vvw	1.836	1	1.833	2	1.828		
2	1.928			vw	1.801			4	1.784		
1	1.905			wb	1.761	2	1.735	1	1.730		
1	1.868	1	1.854					1	1.700		
1	1.843	1	1.833	vvw	1.676			1	1.680		
1	1.836	1	1.819	vvw	1.653			3	1.660		
1	1.783	1	1.783								
1	1.763	1	1.760								
1	1.732										
>1	1.714	1	1.708								
1	1.674										
2	1.657	1	1.649								

*For the Stewart, Chalmers, and Phillips data, the order of decreasing intensities is: vs^1, vs^2, s^3, mw, vw, vvw.

**b indicates broadline.

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HYDROTHERMAL SYNTHESIS OF VEATCHITE AND ITS CALCIUM ISOMORPH, by C. R. Parkerson, Research Department. 1 June 1963. 16 pp.

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2. Strontium borate—Synthesis
3. Veatchite—Synthesis
I. Parkerson, C. R.
II. Title

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