NOBLEITE, ANOTHER NEW HYDROUS CALCIUM BORATE FROM THE DEATH VALLEY REGION, CALIFORNIA*

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ABSTRACT

Nobleite is a new hydrous calcium borate, $CaO \cdot 3B_2O_3 \cdot 4H_2O$, that has been found in the Furnace Creek borate deposits of the Death Valley region, California. Platy euhedral crystals of nobleite are produced by the weathering of colemanite and priceite veins in altered basaltic rocks of the Furnace Creek formation of late Tertiary age. The new mineral is associated with ulexite, gowerite, meyerhofferite, colemanite, several undescribed borates, ginorite, sassolite, a manganese oxide mineral, and gypsum.

Nobleite is biaxial (+), $\alpha = 1.500 \pm 0.003$, $\beta = 1.520 \pm 0.002$, $\gamma = 1.554 \pm 0.002$; $2V = 76°$ (calc.); $r > v$ weak; $X \wedge a = 29°$, $Y = b$, $Z \wedge c = -7°$. Hardness 3; measured specific gravity 2.09 ± 0.01.

Chemical analysis of the mineral gave: Li_2O 0.02, Na_2O 0.26, K_2O 0.06, CaO 16.96, SrO 0.11, Fe_2O_3 0.15, B_2O_3 60.80, H_2O 21.84; total 100.28 per cent.

Nobleite is monoclinic; the space group is $P2_1/a$; $a = 14.56 \pm 0.05$ Å, $b = 8.01_6 \pm 0.02$ Å, $c = 9.83_8 \pm 0.02$ Å, $\beta = 111° 45' \pm 10'$; $a:b:c = 1.816:1:1.227$; cell volume 1066 Å³; cell contents $4[CaO \cdot 3B_2O_3 \cdot 4H_2O]$; calculated density 2.09_8.

The new mineral is named in honor of Dr. Levi F. Noble of the U. S. Geological Survey.

INTRODUCTION

The mineral described in this paper was first collected by McAllister in 1955 in the course of his study of the Furnace Creek borate district in the Death Valley area, California. His preliminary tests showed the material to be a borate having optical properties different from those of other naturally occurring borates. Further study by Erd and Vlisidis proved this material to be identical with the synthetic compound $CaO \cdot 3B_2O_3 \cdot 4H_2O$.

The naturally occurring hydrous calcium borate, $CaO \cdot 3B_2O_3 \cdot 4H_2O$ (or $CaB_6O_{10} \cdot 4H_2O$), is named nobleite in honor of Dr. Levi F. Noble, geologist in the U. S. Geological Survey since 1909, in further recognition of his fundamental contributions to geologic knowledge of the Death Valley region. Dr. Noble's broad interest in the region has included the geology of borates and the paragenetic sequence of colemanite after ulexite in some mines.

OCCURRENCE

Nobleite has been found at seven places that are widely spaced for twelve miles along virtually the length of the area containing borates between the floor of Death Valley and the Widow mines in the Greenwater Range south of Ryan. These localities are at least one mile and

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* Publication authorized by the Director, U. S. Geological Survey.

not more than four miles apart on mining property of the United States Borax and Chemical Corporation. All but the southeasternmost one are within the boundary of Death Valley National Monument. The number of localities indicates little more than how far the search for the new mineral has been carried.

The mineralogical observations in this paper were made on material from a locality 0.6 mile NNW of the De Bely mine or 1.1 miles in a straight line S. 18° E. from the junction of the Corkscrew Canyon road and California highway 190.

The nobleite is associated with other borate minerals produced by the weathering of colemanite and priceite veins in altered olivine basalt and basaltic clastic rocks (McAllister, 1958; Erd and others, 1959, p. 912–913) in the Furnace Creek formation (Noble, 1941, p. 955–956) of late Tertiary age. The new mineral generally is in surficial, weathered material on and near the source veins. The weathered matrix now consists chiefly of montmorillonite, analcime, and some magnetite. It contains along with borate minerals some gypsum, thenardite, a manganese oxide mineral, and limonite. The newly formed borates in surficial material at nobleite localities include ulexite, gowerite, meyerhofferite, colemanite, an undescribed calcium borate, a magnesium borate, ginorite, and sassolite. An apparently exceptional occurrence of the most coarsely crystalline nobleite was found in the Corkscrew mine by Mr. Sutherland, who describes it below. Two other occurrences underground, about 10 feet vertically from the surface in the Hard Scramble claim and 300 feet below the surface at the Widow No. 3 mine, are more similar to the others, for they are in efflorescences on mine workings in priceite- or colemanite-bearing altered basalt.

At every locality where the original colemanite or priceite veins are exposed (six out of the seven places), some of the nobleite encrusts the veins, which generally show corrosion. Some aggregates of subhedral nobleite crystals retain the general form of diverging clusters of the original coarse colemanite. A crust of meyerhofferite in one specimen lies between the colemanite and some of the nobleite. Clusters of gowerite crystals are attached to nobleite aggregates from four of the localities and some of this gowerite in turn supports individual crystals of nobleite (Fig. 1). A still undescribed calcium borate at one place fills in between the microblades of gowerite on the nobleite. Much of the nobleite forms a coherent open meshwork of tabular crystals, commonly intergrown with fluffy ulexite, an undescribed manganese oxide, or doubly terminated microcrystals of colemanite. Like closely associated ginorite and sassolite, clots of euhedral nobleite are also irregularly spaced through a nearly incoherent matrix of weathered basaltic surficial debris.

Some of the ginorite is interspersed among the larger crystals of nobleite, coating them and filling in between as if at least this ginorite formed later than this nobleite.

According to field relationships, the origin of the nobleite in surficial material can be attributed with some confidence to weathering of borate minerals in the Tertiary rocks. How recently it has continued to crystal-lize since the present topography was formed remained unanswered until
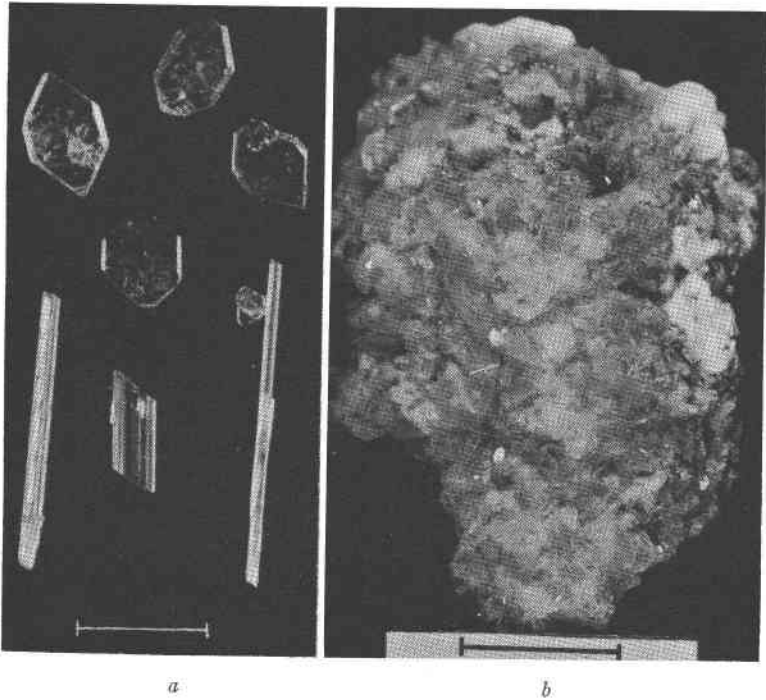

FIG. 1. (a) Crystals of nobleite (at top) and gowerite from north of the De Bely mine, Death Valley. Crystals of nobleite may be seen adhering to the gowerite crystal on the right. Bar at bottom indicates one mm. (b) Nobleite crystals from the Corkscrew mine, Death Valley. Bar at bottom indicates one cm. Photographs by Elliot C. Morris.

nobleite was found in the efflorescence coating man-made surfaces less than forty years old.

The largest best-formed crystals of nobleite (Fig. 1) were obtained from the Corkscrew mine in December 1957, by J. A. Sutherland,[1] who at the time was unaware that the new mineral from nearby localities was being described for publication. He had previously collected a small

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amount of the material in November 1956. Mr. Sutherland contributes the following description of the occurrence of the material he collected:

The nobleite in the Corkscrew mine was found on colemanite in several geodelike cavities which were joined by small fissures. This enabled the tracing of nobleite in this occurrence. The lower cavity was entirely coated with nobleite, while the upper cavities were only partially covered. The lower cavity showed the largest crystals and some of the most perfectly formed. The upper cavities showed smaller crystals of good form, though they are not as translucent as the crystals of the lower cavity. A few isolated crystals were noticed on colemanite in fissures below the lower cavity. The crystals range in size from less than 1 mm. to more than 3 mm. There were only two minerals in direct association with the nobleite, colemanite as mentioned and ulexite. Nobleite appears to be a solution deposition and of later origin than the colemanite and probably earlier than the ulexite. No ulexite was found directly on nobleite but several tufts of ulexite crystals were found in the upper cavities and showed no apparent damage or etching from a later solution.

The Corkscrew mine is 1.6 miles in a straight line S. 20° W. from the junction of the Corkscrew Canyon road and California highway 190, as shown on the topographic map of the Furnace Creek quadrangle.

CRYSTALLOGRAPHY

X-ray data

The crystallography of nobleite, determined by single-crystal x-ray precession techniques, is given in Table 1, together with the calculated and observed densities. The diffractometer powder pattern (Table 2) was indexed from the single-crystal data in Table 1, and all calculated d-spacings are listed in Table 2 for $d \geq 2.500$ Å. Observed powder data are given both for the mineral and for synthetic material. Nobleite shows strong preferred orientation in its x-ray powder pattern due to the platy habit and perfect cleavage parallel to (100); this effect is much less pronounced in the pattern of the synthetic nobleite.

Morphology

Nobleite occurs as a coherent open mesh-work of platy euhedral crystals or as mammillary coatings formed by plates arranged sub-parallel to {100}. Fig. 2 illustrates a typical habit for nobleite crystals. The {100} plate form is commonly elongated [010] or [001]. Large crystals have a hexagonal aspect (Fig. 1a), but smaller crystals are commonly rhomb-shaped by truncating {011} forms. The acute angle of the rhombs, measured with the microscope, is 78° (78°25′, calc., from x-ray data). Common forms observed are: {100}, {001}, {110}, {011}, and {111}. These forms were identified by comparing interfacial angles measured under the microscope with angles calculated from the x-ray cell constants. Contact twinning, with (100) as twin plane and composi-

TABLE 1. CRYSTALLOGRAPHIC DATA FOR NOBLEITE, $CaO \cdot 3B_2O_3 \cdot 4H_2O$*

	Symmetry: monoclinic	
	(1)[†] $P2_1/a$	(2)[†] $P2_1/n$
a	14.56 ± 0.05 Å	14.23 ± 0.05 Å
b	$8.01_6 \pm 0.02$	$8.01_6 \pm 0.02$
c	$9.83_8 \pm 0.02$	$9.83_8 \pm 0.02$
β	$111°45' \pm 10'$	$108°11' \pm 10'$
$a:b:c$	1.816:1:1.227	1.775:1:1.227
Cell Volume	1066 Å³	
Cell Contents	$4[CaO \cdot 3B_2O_3 \cdot 4H_2O]$	
Density (calc.)	2.09_8 g.cm.$^{-3}$	
Specific Gravity (meas.)	$D_4^{20°}0$, 2.09 ± 0.01 (mineral)[a]	
	2.10[b] 2.09[c] (synthetic)	

* Single-crystal x-ray data obtained by Joan R. Clark, U. S. Geological Survey (written communication, 1959); a quartz-calibrated precession camera was used with Mo/Zr radiation, $\lambda MoK_\alpha = 0.7107$ Å, and film measurements were corrected for both horizontal and vertical film shrinkage.

[†] Transformation, $P2_1/a$ to alternative setting $P2_1/n:\overline{1}0\overline{1}/010/001$.

[a] Determined by Erd for clear inclusion-free crystals using the sink-float method with a mixture of bromoform and carbon tetrachloride.

[b] Determined by Vlisidis using a fused silica Adams-Johnston pycnometer with ethyl alcohol (95% by volume) as the liquid medium; synthetic nobleite prepared by W. T. Schaller, U. S. Geological Survey.

[c] Chelishcheva (1940, p. 511) on synthetic material.

tion surface is common. Such twins show symmetrical extinction in sections containing [010]. Typical dimensions of individual crystals from north of the De Bely mine are 0.6 by 0.3 by 0.1 mm. One specimen from the Corkscrew mine contains crystals up to 3 mm. in length, covered with a dust of later nobleite crystals having an average diameter of 0.01 mm.

PHYSICAL AND OPTICAL PROPERTIES

Nobleite exhibits perfect {100} and indistinct {001} cleavages; the fracture is uneven. The mineral is sectile, flexible in thin crystals or cleavage flakes, and inelastic. The hardness is about the same as that of calcite (3).

Individual crystals of nobleite are colorless and transparent, but fine-grained aggregates and the streak of the mineral are white. The luster is subvitreous, but is pearly on cleavage plates. Nobleite is neither fluorescent nor thermoluminescent.

TABLE 2. X-RAY DIFFRACTION DATA AND UNIT CELL OF NOBLEITE

Indexed on monoclinic unit cell: $P2_1/a$; $a = 14.56 \pm 0.05$ Å, $b = 8.01_6 \pm 0.02$ Å, $c = 9.83_8 \pm 0.02$ Å, $\beta = 111°45' \pm 10'$.

hkl	Nobleite North of De Bely mine, Death Valley[1]			$CaO \cdot 3B_2O_3 \cdot 4H_2O$ Synthetic[2]	
	d (calc.)[3] Å	d (meas.) Å	I	d (meas.) Å	I
001	9.138				
110	6.897				
$\bar{2}01$	6.766⎫	6.79	100	6.78	100
200	6.764⎭				
$\bar{1}11$	6.086⎫	6.06	3	6.05	5
011	6.027⎭				
$\bar{2}11$	5.171⎫	5.18	9	5.18	20
210	5.170⎭				
111	5.064				
$\bar{2}02$	4.674⎫	4.68	5	4.67	9
201	4.671⎭				
002	4.569	4.58	2	4.57	6
$\bar{1}12$	4.191				
$\bar{3}11$	4.126				
$\bar{2}12$	4.038⎫	4.05	3	4.04	7
211	4.036⎭				
020	4.009				
012	3.970				
310	3.930	3.94	5	3.93	8
120	3.844			3.83	3
$\bar{1}21$	3.684				
021	3.671⎫	3.65	2	3.66	3
$\bar{4}01$	3.641⎭				
$\bar{3}12$	3.611				
112	3.515	3.51	1	3.51	2
220	3.449⎫	3.45	5	3.45	22
$\bar{2}21$	3.449⎭				
121	3.417				
$\bar{4}02$	3.383⎫	3.39	31	3.39	12
400	3.382⎭				

(continued on next page)

[1] Radiation: Fe, unfiltered; only lines due to $\lambda FeK_\alpha = 1.9373$ Å radiation are listed. Shows preferred orientation so that intensities shown are not directly comparable with those of the synthetic compound. Diffractometer chart X-493.

[2] Radiation: Cu/Ni, $\lambda CuK_\alpha = 1.5418$ Å. Diffractometer chart X-1456. Synthetic material prepared by R. C. Erd.

[3] All calculated spacings listed for $d_{hkl} \geq 2.500$ Å.

TABLE 2. (*continued*)

	Nobleite North of De Bely mine, Death Valley[1]			$CaO \cdot 3B_2O_3 \cdot 4H_2O$ Synthetic[2]	
hkl	d (calc.)[3] Å	d (meas.) Å	I	d (meas.) Å	I
$\overline{4}$11	3.315				
$\overline{2}$03	3.267	3.27	2	3.27	4
202	3.266				
311	3.249				
$\overline{4}$12	3.117				
410	3.116	3.12	7	3.11	12
$\overline{1}$22	3.107				
$\overline{3}$21	3.080	3.08	4	3.08	10
003	3.046				
$\overline{2}$22	3.043	3.05	3	3.05	10
221	3.042				
$\overline{2}$13	3.026				
212	3.025				
022	3.013				
$\overline{1}$13	3.004	3.006	5	2.998	14
320	2.996				
$\overline{3}$13	2.904			2.912	3
$\overline{4}$03	2.848				
401	2.847	2.849	5	2.849	15
$\overline{3}$22	2.847				
013	2.847				
122	2.800	2.805	2	2.801	7
$\overline{5}$11	2.730	2.734	1		
$\overline{4}$21	2.695				
$\overline{4}$13	2.684	2.685	1	2.691	2
411	2.683				
$\overline{5}$12	2.671				
321	2.660				
130	2.622	2.614	1	2.622	2
113	2.610				
312	2.594				
420	2.585				
$\overline{4}$22	2.585				
$\overline{1}$31	2.570				
031	2.565	2.566	9	2.567	13
510	2.563				
$\overline{2}$23	2.533				
222	2.532				
$\overline{1}$23	2.520				
		2.489	1	2.481	3
		2.464	1	2.462	3
		2.410	5	2.413	3

TABLE 2. (*continued*)

hkl	Nobleite North of De Bely mine, Death Valley[1]			CaO·3B₂O₃·4H₂O Synthetic[2]	
	d (calc.)[3] Å	d (meas.) Å	I	d (meas.) Å	I
		2.309	7	2.304	9
		2.256	4	2.246	5
		2.171	4	2.171	3
		2.119	2		
		2.098	5	2.096	23
		2.067	2	2.060	8
		2.022	2	2.019	6
		1.986	3	1.985	12
		1.963	5	1.965	6
		1.905	1	1.903	4
		1.881	1	1.881	2
				1.848	2
		1.831	0.5	1.831	4
		1.805	1	1.807	3
		1.788	0.5	1.787	2
		1.744	2	1.743	4
		1.721	1	1.724	3
		1.692	4	1.692	2
		1.636	0.5	1.637	1
		1.615	0.5	1.613	1
				1.585	1
				1.547	1
		1.521	0.5	1.519	1
	plus additional lines all with I≤2				

Nobleite is colorless in all orientations in transmitted light. It is biaxial (+) with the following optical properties:

	n	
α	1.500 ± 0.003	$2V = 76°$ (calc.)
β	1.520 ± 0.002	$r > v$, weak
γ	1.554 ± 0.002	$X \wedge a = 29°$, $Y > b$, $Z \wedge c = -7°$

Nikolaev and Chelishcheva (1940, p. 129) report $\alpha = 1.505$ and $\gamma = 1.552$ for the synthetic compound.

CHEMICAL PROPERTIES

Analysis

Large (1 mm. in diameter) and nearly inclusion-free crystals of nobleite were selected for analysis. The sample, weighing 0.500 g., was dissolved with 30 ml. of 1 N HCl. This solution was adjusted to a pH of 2.5 and
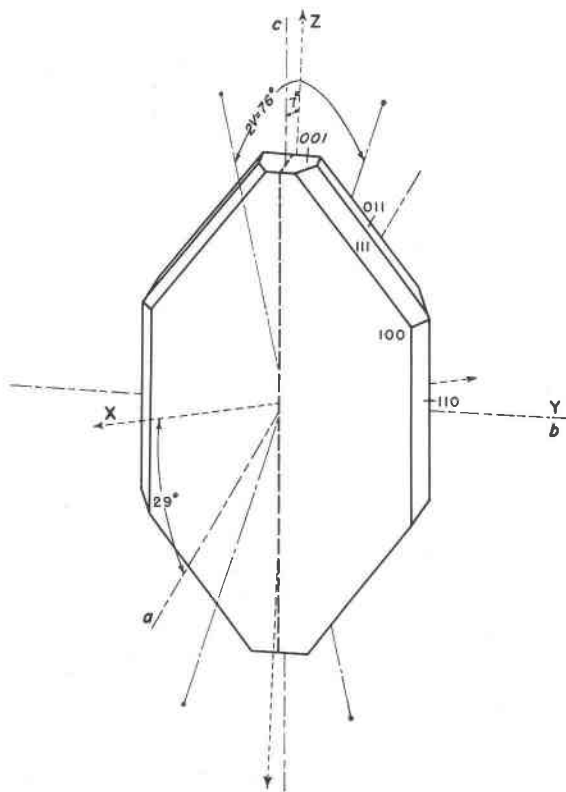
FIG. 2. Typical habit and optical orientation of nobleite. Interfacial angles are approximate.

then passed through an ion exchange column in order to separate B_2O_3 from the cations. Mannite was added to the effluent and B_2O_3 was titrated with NaOH in the usual way. The cations were recovered from the column and determined by the standard methods of quantitative analysis as outlined by Hillebrand and others (1953). Total water was measured by the Penfield method.

The results of the analysis are given in Table 3.

Synthesis

The compound $CaO \cdot 3B_2O_3 \cdot 4H_2O$ was first reported by Ditte (1873, p. 784) who prepared it by dissolving $CaCO_3$ in a boiling concentrated solution of boric acid. Its synthesis was subsequently reported by Meyerhoffer and Van't Hoff (1907), Van't Hoff (1907, p. 653), Sborgi (1913, p. 640–641), Nikolaev and Chelishcheva (1940), and Kurnakova (1953, p. 46). Dr. W. T. Schaller, who has been working for a long period

TABLE 3. CHEMICAL ANALYSIS OF NOBLEITE

	Weight per cent*	Molecular proportions	Ratios	Calculated composition for $CaO \cdot 3B_2O_3 \cdot 4H_2O$
CaO	16.96	.3024 ⎫ .3035	1.01	16.64
SrO†	0.11	.0011 ⎭		
B_2O_3	60.80	.8731	2.92	61.98
H_2O	20.82 ⎫ 21.84	1.2123	4.05	21.38
H_2O^-	1.02 ⎭			
Fe_2O_3	0.15			
Na_2O†	0.26			
K_2O†	0.06			
Li_2O†	0.02			
Insoluble residue	0.08			
	100.28			100.00

* A. C. Vlisidis, analyst.

† Determinations made by Leonard Shapiro using a Beckman Model DU flame spectrophotometer.

of time on the synthesis of calcium borates (as a part of a larger study on borates), has prepared this compound in various ways (personal communication, 1958).

The fine-grained nobleite (to 4 μ in diameter) used in obtaining the *x*-ray powder data reported in Table 1 was synthesized by Erd as follows: 1.6 g. CaO and 10.0 g. H_3BO_3 were stirred in 25 ml. H_2O for 30 hours at 48° C.; the resulting product was held at 68° C. for 10 days; finally the precipitate was filtered, washed successively with distilled water, methyl alcohol, and acetone and air-dried at room temperature.

Solubility data

Nobleite is slightly soluble in cold water; moderately soluble in hot water. Nikolaev and Chelishcheva (1940, p. 128) in their study of the solubility isotherm of the system CaO—B_2O_3—H_2O at 25° C. found that after 153 days the composition of the liquid phase in equilibrium with nobleite (as the only solid phase) was B_2O_3 1.34, CaO 0.044, (and H_2O 98.616) per cent. Calculation of the CaO as nobleite shows 2.71 g. of nobleite and 21.4 g. of boric acid per liter of H_2O to be in solution under equilibrium conditions. Although not directly comparable, this solubility of nobleite in a solution of boric acid may be contrasted with the solubility of gowerite in water = 6.03 g./liter (Gode, 1949).

The mineral dissolves readily in cold dilute acids and strong alkalies;

is slowly soluble in a 30 per cent solution of hydrogen peroxide, in xylol, and in glyerol; and is very slightly soluble in methyl alcohol. Nobleite reacts with dilute sulfuric acid to produce crystals of gypsum and boric acid.

Pyrognostics

Before the blowpipe, nobleite decrepitates and then fuses to an opaque enamel bead. In the closed tube test, nobleite decrepitates strongly, exfoliates slightly, and finally becomes a frothy glass. The water driven off during the heating has a pH of about 3 indicating a loss of B_2O_3.

Rapidly heated to 465° C. in an electric furnace, crystals of nobleite were found to have exfoliated perpendicular to {100} and to have turned silvery white. Outlines of the original crystals are still sharp at this temperature; however, most of the material has become a glass ($n = 1.558$) with many inclusions rendering it almost opaque. With continued heating the glass softens at about 580° C. and melts completely by 610° C.

Chemical nomenclature and relationships

The name "triborate" was first applied to the compound $CaO \cdot 3B_2O_3 \cdot 4H_2O$ by Meyerhoffer and Van't Hoff (1907, p. 104) and this term has been used by subsequent workers (for example, Kurnakova, 1953) to refer to this compound and, in a general sense, to the class of borates having a ratio of $B_2O_3/MO = 3$. The equally established term "hexaborate" (derived from the empirical formula $MB_6O_{10} \cdot xH_2O$) has been used (Gode, 1949; Gode and Kesans, 1953) to refer to the same class of borates and to $CaO \cdot 3B_2O_3 \cdot 5H_2O$. However, crystal chemistry provides a logical and systematic basis for classification of hydrated borates (Christ, 1960). The crystal structure of gowerite $CaO \cdot 3B_2O_3 \cdot 5H_2O$, is currently being studied (Christ and Clark, 1960) together with those of other members of the $CaO \cdot 3B_2O_3 \cdot xH_2O$ series (Christ and Clark, written communication, 1959). Further studies by one of us (R. C. Erd), using differential thermal analysis, dehydration data, and x-ray powder techniques, are in progress in order to elucidate the relationships of the members of this series to one another and to other hydrated borates. The results obtained to date indicate that there is no simple hydration-dehydration relationship between nobleite and gowerite.

Geological Survey. Especially, we are indebted to Joan R. Clark for her x-ray study of the single crystals and for a most helpful critical review of this paper; to W. T. Schaller for many helpful discussions and for the loan of synthetic nobleite specimens, to C. L. Christ for much useful advice, and to Daniel E. Appleman who carried out the computation of the d-spacings on a digital computer.

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Manuscript received June 30, 1960.