

A NEW BORATE MINERAL FROM THE UPPER LOUANN FORMATION IN THE NORTHERN GULF

Delbert E. Gann Department of Geology, Millsaps College

> David T. Dockery III Mississippi Bureau of Geology

> > John C. Marble Jackson, Mississippi

ABSTRACT

An apparently undescribed mineral of the Hydrated Borate Group was found in the upper Louann Formation at its boundary with carbonates of the overlying Smackover Formation in two deep wells in Smith County, Mississippi, and in a third well outside of Mississippi. X-ray diffraction data for this mineral show the closest approximation to be with the Borate Group mineral called Ericaite, a mineral closely related to Boracite. This new borate occurs as clear, sand-sized, pseudo-isometric crystals with a hardness between 6 and 7. Apart from their euhedral isometric form, these crystals could readily be mistaken for quartz sand. It is probable that this new borate mineral occurs in residual deposits at the top of the Louann Formation due to dissolution of salt at its upper boundary.

INTRODUCTION

A new borate mineral having sand-sized, clear, pseudo-isometric crystals was found in three wells



Figure 1. Borate crystals (cubes and tetrahedrons) from the American Quasar Petroleum and Conwood Exploration, #1 Beatty 1-14, Smith County, Mississippi. penetrating evaporites of the upper Louann Formation (Figure 1). Two of these wells are the American Quasar and Conwood Exploration, #1 Beatty 1-14, and the Shell et al., #1 Orin C. James, both in Smith County, Mississippi. Samples containing the new borate mineral were submitted by a sample service from a third well outside of Mississippi. The name and location of this well were not released, but the interval containing borate crystals was from a similar depth as those in Smith County and the stratigraphic interval is assumed to be the same.

The American Quasar and Conwood Exploration, #1 Beatty 1-14, was a wildcat well drilled to a total depth of 17,396 feet reportedly in Louann salt. In a review of the logs of this well by one of us (JCM), it was not readily apparent from the wireline logs that the salt had indeed been reached. Further comparison of these wireline logs with the sample log (Figure 2) indicated that a sandstone may have been drilled in the last thirty feet.

A follow-up conversation with the well-site geologist indicated that the reasons for reporting salt were an increase in penetration rate while drilling, and an increase in chloride content of the mud. Admittedly, the samples were of poor quality, but there were apparently enough samples and such a change in lithology that the mudlogger documented a change from dense limestone in the lower Smackover to a sandstone. If this lower unit were indeed a sandstone, then it might be the Jurassic Norphlet Sandstone or a sandstone developed in the lower Smackover. In any event, a correct determination of the bottom-hole lithology of this well was important in evaluating the petroleum potential of the area.

Cuttings from the #1 Beatty well on file at the Mississippi Bureau of Geology were examined and the prominent lithologic change at the base of the Smackover was noted. Samples from the basal unit of this well consisted of light tan and gray, finegrained cuttings and clear sand-sized grains with euhedral faces. Many of the clear grains had the form of cubic crystals. These crystals lacked a salty taste, were hard, and did not dissolve in water, so they obviously were not halite crystals. Other isometric crystals of tetrahedrons and cubes modified by tetrahedrons were also present (see Plate 1). It appears that these crystals led the mudlogger to call the lower unit a sandstone. Apart from being apparently isometric, these crystals exhibit the clarity and hardness of quartz sand grains.

X-ray diffraction analyses at the Geology Department, Millsaps College, determined these crystals and the associated fine-grained matrix to be a borate, though the d-spacings did not fit that of any previously named borate species. In physical appearance and hardness this new borate resembles Boracite. According to Palache et al. (1951, p. 380), Boracite is an evaporite that occurs in association with halite, anhydrite, and gypsum. It is found at Stassfurt and other localities in Germany and has been observed in the insoluble residues of salt wells in Louisiana.

STRATIGRAPHIC SETTING

The three wells in which the new borate mineral was found penetrated the lower Smackover limestone and reached total depth in the top of the Louann Formation. Cuttings containing the borate in the #1 Beatty and #1 O.C. James wells were in the 17,380-17,397 and 17,060-17,075 feet samples, respectively. In the case of the #1 Beatty, it was important to discern whether the borate was an in situ evaporitic phase of the Norphlet or was part of an evaporite sequence above the Louann salt. As no terrigenous sediments (i.e. quartz sand) were associated with the borate in the samples, it was assumed that the evaporite sequence is associated with the Louann salt.

The occurrence of borate-containing evaporites at the top of the Louann salt poses the question as to whether these evaporites represent an in situ evaporitic sequence above the salt or represent a residual or cap rock from leaching of the salt sequence at its upper boundary. The latter opinion was proposed by Theo Dinkins (personal communication), who has seen clear borate crystals above the Louann salt where it was in contact with various stratigraphic units due to salt movement. This is also the opinion of the writers. According to Dinkins, the occurrence of borate crystals above the Louann salt is widespread in Mississippi. They are probably overlooked by many geologists who give them little notice and possibly record them as quartz sand or consider them to be contamination from uphole. In the #1 Beatty well, the thick overlying carbonate sequence of the Smackover precludes the likelihood that the borate crystals would be mistaken as quartz sand from uphole contamination. As geologists working in the northern Gulf region become aware of this new borate mineral, its occurrence will be better documented.

CRYSTALLOGRAPHY OF THE NEW BORATE MINERAL

All crystals of the new borate mineral selected from the cuttings exhibit exceptionally well-formed euhedral faces, suggesting in situ (authigenic) crystal growth. The forms examined display



Figure 2. Modified basal section of sample log for the #1 Beatty 1-14. The lower unit described as a "sand" contains borate crystals.



Figure 3. Some of the larger cubic borate crystals from the #1 Beatty 1-14. The cubic crystal in the upper right is still embedded in the associated fine-grained, light tan matrix. A cluster of pyrite crystals is attached to the face of the cube.

symmetry elements compatible with the isometric crystal system. As will be demonstrated later, however, this point is somewhat moot, since this mineral species very likely represents replacement of some pre-existing mineral phase.

It is important here, however, to adequately demonstrate that these crystal forms are indeed representative of the isometric system. This system includes a number of symmetry classes and forms. The crystals examined here have three crystallographic axes of 4-fold rotoinversion. There are four diagonal axes of 3-fold rotation, and six diagonal mirror planes. This symmetry is symbolically noted as $3\overline{A}_A$, 4A₂, 6m. There are four forms in this class, including the hextetrahedron. The hextetrahedron has 24 faces that can be seen as a tetrahedron, cube, dodecahedron, and tristetrahedron. As illustrated in Plate 1, several of these modifications and combinations of modifications are represented by the crystals examined for this report. These forms are compatible with crystals in the Hextetrahedral Class of the Isometric System.

MINERALOGY

Physical Properties

Most of the crystals collected are 1 mm or less in diameter. Therefore, determinations of some of the physical properties proved difficult and likely represent values containing small measurement errors, especially with respect to hardness and specific gravity.

Hardness: Ordinary scratch tests were performed on known mineral surfaces using the unidentified crystals as the scratching tool. Potash feldspar (hardness = 6) was easily scratched by the unknown. On the other hand, quartz (hardness = 7) was not visibly marked using the same procedure. Therefore, the best estimate of the hardness of the unidentified mineral is placed between 6.2 and 6.8.

Specific Gravity: The specific gravity (density could not be determined at this point because the dimensions of the unit cell are not yet known) was determined by weighing a group of the crystals in air and then in water. The specific gravity was then calculated using the relationship:

where Wa represents the weight of the crystals in air and Ww represents the weight of the crystals in water. This calculation suggests the specific gravity of the unidentified mineral is approximately 2.88.

Color, Streak, Luster, and Transparency: All of the crystals examined, with one exception, are colorless. One crystal was very pale green. The streak is white (i.e. when the mineral is powdered). The luster is at least vitreous, but many crystals approach adamantine. Most of the crystals are clear and transparent with no recognizable inclusions.



Figure 4. An assortment of borate crystals from the #1 Beatty 1-14.

Some of the crystals have been etched slightly and present frosted to pitted faces, causing them to appear translucent. This is especially true of samples from the out-of-state well. Most of these latter samples appear also to have been slightly dissolved so that many of the crystal edges and corners are somewhat rounded.

Twinning: Penetration-type twins are common. They are represented both by cubic and tetrahedral forms. Several of these twins are illustrated in Figure 3. Some of the crystals observed with the aid of the petrographic microscope show a crude parallel arrangement of laths, alternatingly extinct and illuminated under crossed polarizers during stage rotation. Although somewhat similar to polysynthetic twinning, this phenomenon could also be explained as the result of crystal strain due in part to the depth of burial (17,000+ feet).

Cleavage: Cleavage could not be verified with authority, but several crystals were ruptured with a steel probe (dissecting needle) without producing any recognizable or reproducible cleavage pattern. It is assumed that this mineral does not possess cleavage. On the other hand, conchoidal fracture is common.

Optical Properties

A large number of the unidentified crystals were ground by hand to an appropriate size for investigation by means of the petrographic microscope. The color, anisotropism, optic sign, and refractive indices were then determined from this powder in a preliminary fashion.

Anisotropism: The grains are colorless in plane polarized light and are neither pleochroic nor absorbtive, nor do they show any bireflecting characteristics. The crushed grains also do not reveal any distinctive cleavage patterns. Under crossed polarizers the grains are observed to be anisotropic. Anisotropism is not generally compatible with isometric crystals, which are isotropic in character. Under conditions of deep burial, however, isometric crystals can be subject to strain of the crystallographic/optic axes and thus present crystals that have properties of anisotropic crystals. As indicated earlier in this report, these crystals were obtained from depths exceeding 17,000 feet. This depth of burial could provide the necessary stress and temperature to produce such anomalies. In addition, most of the crystals examined optically



Figure 5. Various crystal forms exhibited by the new borate mineral.

exhibit strong undulose extinctions which can often be diagnostic of strain along the crystallographic axes. On the other hand, if the unidentified mineral is "naturally" anisotropic, then it would seem reasonable that this mineral has pseudomorphed a previously crystallized isometric form as suggested earlier. Palache et al. (1951, p. 378) suggest such a phenomenon for a mineral of the Borate Group. They indicate that Boracite (Mg3B7013CI) is isometric hextetrahedral above 265° C (High Boracite), but at normal temperatures it changes to paramorphic isometric crystals with orthorhombic pyramidal internal characteristics (Low Boracite). This phenomenon is also a likely possibility for the crystals investigated in this report. There is also very strong evidence to suggest that this mineral is indeed a member of the Boracite Group, as discussed later in this report.

Interference Figure: For those grains which are clearly discernible, the interference figure is biaxial with a moderate 2V angle. A grain has not as yet been discovered in an orientation such that both optic axes are present within the field of view, so measurement of the 2V angle has not been possible. Some of the grains are biaxial positive, others are negative. This is probably due to the close proximity of the three indices of refraction and the small birefringence (see below). The birefringence is weak (0.006 - 0.009 Mu). Most grains do not have retardation colors exceeding straw yellow of the first order, and therefore are about the same as for guartz.

Refractive Indices: The refractive indices of the unidentified species were determined in the traditional way by comparing the mineral fragments to various immersion oils of a known refractive index. All three indices, X (alpha), Y (beta), and Z (gamma), are very close to each other as suggested by the weak birefringence. These values have been determined as follows:

$$X = 1.644$$

 $Y = 1.648$
 $Z = 1.652$

The physical and optical data are summarized in Table I.

X-RAY DIFFRACTION

Three types of material from two well locations were examined by means of x-ray diffraction procedures. These materials included unidentified crystals from the #1 Beatty well, crystal matrix from the same well, and crystals submitted from another well described as "out-of-state". The exact location of the latter well was not released for proprietary reasons. Each of the three materials was ground in acetone with the aid of a mortar and pestle, and then the acetone-powder solution was dropped onto a glass slide and allowed to evaporate. Each sample was then scanned with a General Electric XRD-6 xray diffraction apparatus from 3-60° 2-theta, using CuK-alpha radiation. Each of the resulting diffractograms are interpreted and are reported as dspacings and relative intensities in Table II.

It is immediately obvious from the preliminary data reported in Table II that the crystals from the #1 Beatty well and the out-of-state well are the same mineral species, and that the mineral composition of the #1 Beatty matrix material represents a second mineral species. A search through the JCPDS Powder Diffraction File, the literature, and other

Plate 1. Figures 1-3, cubes from #1 Beatty x 20. Figure 4A-B, twinned cubes from #1 Beatty, x 20. Figures 5-13B, combinations of cube and tetrahedron from #1 Beatty, x 20. Figures 14A-E and 15A-E, combinations of cube and tetrahedron from #1 O.C. James showing various views, focuses and lightings, x 40. Figure 16, tetrahedron from #1 O.C. James, x 40. Figure 17, tetrahedron from #1 Beatty, x 20. Figures 18-19, tetrahedron and tristetrahedron from #1 Beatty, x 20.



private sources did not reveal a completely satisfactory match for the unidentified crystals, but some data were close enough to suggest the mineral family to which these crystals and matrix belong. The closest approximation for the crystals is an orthorhombic mineral from the Borate Group called Ericaite (Fe_{2.25}Mg_{0.57}Mn_{0.18}) (B₇0₁₃) Cl, which is apparently closely related to Boracite (Mg₃B₇0₁₃Cl) with manganese and iron commonly substituting for magnesium in the crystal lattice. Compare the data in Table III with that in Table II.

It will be noted that all but the 4.31Å peak of Ericaite are agreeable with the d-spacings of the unidentified crystals. The missing peak could be explained by a lower intensity, or the unidentified crystals could represent a new mineral species not yet described in the literature, or possibly a solid solution series phase between two end members of the Boracite family. The indices of refraction for Ericaite (approximately 1.75) are not compatible with the mineral being considered in this report. Further investigation concerning this mineral is pending.

The matrix material from the #1 Beatty well is closely related to the Hilgardite $Ca_8(B_60_{11})_3Cl \cdot 4H_20$, Parahilgardite $Ca_8(B_60_{11})_3Cl \cdot 4H_20$, CI-Tyretskite $Ca_2B_50_8Cl(OH)_2$ "trimorphous" group. Compare the data in Table II with that of Table III. Most of the "missing" peaks from the unidentified diffraction pattern are in part readily explained by the numerous reflections, doublets, and broad multiple reflections which shroud the "missing" peaks. Again, however, the similarities are too great to dismiss to coincidence. Further investigation into the nature of this mineral is also pending.

CHEMISTRY

Approximately 2 grams of the crystals from the #1 Beatty and the "out-of-state" well were ground and then sent to CSMRI-Analytica in Golden, Colorado, for chemical analyses. These results are reported in Table IV.

A quick appraisal of the data in Table IV reveals a major discrepancy in the percentages reported. The chemical components total 59.15%, which is far short of the 100% anticipated. There are four possible explanations for the difference: (1) analytical error, (2) LOI (loss on ignition), (3) the presence of a large amount of water, or (4) one or more of the above. Since the chemical analyses were duplicated and errors minimized with the use of chemical standards, we can assume that analytical error is not a major factor. LOI and water losses seem to be acceptable explanations for the low totals reported from the chemical analyses. The implication is that this mineral is a mineral species composed of significant magnesium, boron, oxygen, and chlorine with minor amounts of sodium, aluminum, silicon, phosphorus, potassium, calcium, titanium, manganese, iron, and barium. Presumably these minor elements are present in the mineral's crystal lattice because of ionic substitution.

A recalculation of the chemical analyses given above when compared to the idealized formula for Boracite (Mg3B7013CI), yields the results reported in Table V. The recalculation of the analyses of the crystals investigated for this report assumed that the bulk of the LOI was in the form of "oxygen". Using this assumption, the #1 Beatty and "out-of-state" well crystals yield a composition very similar to that of the idealized Boracite composition. The best reasoning at this time assumes the unknown mineral to be a member of the Borate Group, and more specifically the Boracite Family. Although the indices of refraction and hardness of Low Boracite are similar to the mineral investigated in this report, the x-ray diffraction data are distinctively different. Additionally, High Boracite is isotropic. A better interpretation of the composition will be forthcoming after the determination of the unit cell dimensions.

CONCLUSIONS

The physical properties (including hardness, specific gravity, color, streak, luster, transparency, and cleavage), optical properties (including anisotropism, interference figure, optic sign, and indices of refraction), x-ray diffraction data, and chemical analyses suggest that the same mineral recovered from two deep wells, one in Mississippi and a second located "out-of-state", is a member of the Hydrated or Hydroxylated Borate Group. The data do not fit exactly with that of any known mineral or any variety, suggesting that this is a new mineral species, or at least not adequately described as a member of the Borate Family solid solution series similar to Ericaite and Boracite. The investigation and study of this mineral is continuing.

REFERENCE CITED

Palache, Charles, Harry Berman, and Clifford Frondel, 1951, The System of Mineralogy of James Dwight Dana and Edward Salisbury Dana, Yale University 1837-1892: 7th ed., v. 2, John Wiley and Sons, Inc., New York, 1124 p.

TABLE I

SUMMARY OF PHYSICAL AND OPTICAL DATA

COLOR	STREAK	LUSTER	HARDNESS	SP.G.	TWINNING	CLEAVAGE
Colorless	White	Vitreous- Adamantine	6.2-6.8	2.88	Penetration- Type	absent
	BIREFRI	NGENCE OPT	C SIGN REF	RACTIVE	INDICES	
	0.006 -	0.009 (+)	or (-)	X = 1.6 Y = 1.6 Z = 1.6	44 48 52	

TABLE II

X-RAY DIFFRACTION DATA OF WELL MATERIALS

NO. 1 BEATTY (CRYSTALS) "OUT-OF-STATE" (CRYSTALS) NO. 1 BEATTY (MATRIX)

1.959

D-Spacing A	Intensity %	D-Spacing A	Intensity %	D-Spacing A	Intensity %
6.05	50	6.05	20	5.73	30
3.490	50	3.496	60	5.64	30
3.017	100	3.015	100	5.01	10
2.704	75	2.701	65	4.18	15
2.474	30	2.465	25	3.90	5
2.135	35	2.135	40	3.17	40
2.043	80	2.042	90	3.14	30
1.823	20	1.820	25	3.12	15
1.744	15	1.743	20	3.097	15
1.676	10	1.674	10	3.066	35
1.615	5	1.613	10	2.873	100
1.573	5	1.572	10	2.820	85
				2.746	30
				2.620	10
				2.589	10
				2.503	10
				2.473	15
				2.442	10
				2.409	10
				2.137	15
				2.116	30
				2.102	15
				2.074	10
			24	2.026	20
				1,992	10

25

TABLE III

X-RAY DIFFRACTION DATA FOR ERICAITE AND CL-TYRETSKITE

ERICAIT	E	CL-TYRETSKITE			
D-Spacing A	Intensity %	D-Spacing A	Intensity %		
6.07	10	5.756	35		
4.31	20	5.652	30		
3.51	40	5.035	30		
3.043	100	4.998	5		
2.720	60	4.918	3		
2.487	15	4.161	20		
2.152	45	3.927	1		
2.056	70	3.200	19		
2.027	15	3.182	40		
1.925	5	3.167	8		
1.834	40	3.147	35		
1.756	25	3.112	17		
1.687	20	3.079	10		
1.626	15	3.066	30		
1.582	5	2.880	100		
		2.827	85		
		2.767	35		
		2.757	55		
		2.742	45		
		2.630	15		
		2.600	10		
		2.530	3		
		2.520	10		
		2.509	8		
		2.498	3		
		2.482	12		
		2.458	8		
		2.415	8		
		2.152	10		
		2.145	10		
		2.130	30		
		2.120	30		
		2.112	8		
		2.104	13		
		2.037	6		
		2 039	14		
		2 029	65		
		2 012	10		
		1 997	16		
		1 963	35		
		1.955	1		
		1.947	15		
		420307636215200	1000 C		

TABLE IV

X-RAY FLUORESCENCE (XRF) RESULTS OF NO. 1 BEATTY AND "OUT-OF-STATE" CRYSTALS

					C	ompone	nt					
Na ₂ 0	Mg0	Al ₂ O ₃	Si02	P205	CI	к ₂ 0	Ca0	Ti0 ₂	Mn0	Fe ₂ 03	Ba0	В
					٧	Weight %	6					
0.21	31.8	0.34	0.77	0.01	5.53	0.04	0.59	0.02	0.01	0.47	0.17	19.2

TABLE V

CHEMICAL COMPARISON OF BORACITE AND THE NO. 1 BEATTY/"OUT-OF-STATE" CRYSTALS

			Composition of				
Boracite Composition (Wt. %)		sition (Wt. %)	No. 1 Beatty/"Out-of-State" Crystals (Wt. %				
Mg	÷	18	Mg	-	19.0		
B	8	20	B	÷	19.2		
0	-	53	0	-	56.2		
CI	-	9	CI		5.5		

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