

X-RAY STUDY OF LECONTITE

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ABSTRACT

Precession photographs of synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ indicate that it belongs to space group $P 2_12_12_1$. Powder photographs of this material are very similar to those for natural lecontite $\text{Na}(\text{NH}_4, \text{K})\text{SO}_4 \cdot 2\text{H}_2\text{O}$. The specimens examined and the unit cell edges upon which their powder photographs could be indexed are:

	<i>a</i>	<i>b</i>	<i>c</i>
$\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$	8.23	12.88	6.26 Å
Lecontite, U.S.N.M. R6085	8.24	12.85	6.24 Å
Lecontite, Yale Coll. 1696	8.23	12.86	6.25 Å
Lecontite, Yale Coll. 4863	8.24	12.88	6.24 Å

Although the lines in the powder pattern of synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ could be entirely indexed on the cell cited, patterns of natural lecontite possessed several lines which could not be indexed. These unindexed lines, however, coincide with the strong lines of thenardite, mascagnite and/or arcanite and are probably attributable to the admixture of these materials in most natural lecontite.

The samples of natural lecontite at the writers' disposition were so fine-grained as to preclude single crystal studies. However, the optical data reported by Palache *et al.* (1951, p. 438) as well as the powder diffraction data are so similar to the data for synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ that the two are probably isostructural. If so, the crystal class for lecontite should be 222 rather than the currently cited $2/m 2/m 2/m$.

Chemical analyses of lecontite may be in error as to potassium, ammonium, and/or sodium to the extent that arcanite, mascagnite, or thenardite are present but unrecognized. The density of the synthetic crystals was measured as 1.745 and calculated to be 1.737 on the basis of $Z=4$.

INTRODUCTION

Unindexed powder patterns have been reported for natural lecontite $\text{Na}(\text{NH}_4, \text{K})\text{SO}_4 \cdot 2\text{H}_2\text{O}$ by Winchell and Benoit (1951, p. 598) and for synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ by the American Society for Testing Material's card 2-0161 which somewhat tentatively equates the synthetic material to lecontite. The present study was undertaken to provide unit cell and space group data, to index the powder patterns, and to confirm or deny the identity of lecontite and synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ crystals.

Reported occurrences of natural lecontite have been confined to Central America. The mineral is found in bat guano, and most specimens have come from a cave near Las Piedras, Comayagua, Honduras.

EXPERIMENTAL WORK

Materials. Samples of lecontite were obtained from the U. S. National Museum and Yale University with catalogue numbers and localities as follows:

U. S. National Museum

No. R6085, Las Piedras, Comayagua, Honduras

Yale University

No. 4863 (Series III), Comayagua, Honduras

(Vaux Coll. No. 15337)

Nos. 1696, 1697 (Series III), Taylor's cave of Las Piedras, Comayagua, Honduras

(1697 has no "original label")

Crystals of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ were grown from the evaporation at room temperature of aqueous solutions of sodium sulfate and ammonium sulfate, the latter in excess of its stoichiometric ratio. The crystals thus produced were separated by means of their optical properties from the crystals of synthetic mascagnite, also precipitated in the beakers. In this way clear single crystals of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$, whose optical and physical properties agreed closely with those quoted for lecontite (Palache *et al.*, 1951, p. 438), were selected for study.

Diffraction data. None of the samples of natural lecontite contained crystals large enough for single crystal studies. All, in fact, were so fine-grained that no grinding was necessary for making their powder patterns. Thus, single crystal photographs were possible only for the synthetic crystals. Precession photographs of several levels, using a^* and b^* as the precession axes, revealed the following conditions for Bragg reflections: hkl , hkO , hOl , and OkI , no conditions: hOO , $h = 2n$; OkO , $k = 2n$; and OOl , $l = 2n$. From these conditions it is concluded that the space group is $P 2_12_12_1$ for synthetic crystals of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$.

The precession photographs, indexed on the basis $c < a < b$, were then used to index a powder pattern of artificial $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$. Indices were assigned to the powder pattern by comparing the 2θ values and intensities of its lines with those of the indexed reflections on the precession photographs, the nomogram of Bloss and Gibbs (1961, p. 31) permitting the 2θ values for the indexed spots on the precession photographs to be quickly determined. After several reflections were so indexed on the powder pattern, the unit cell edges could be calculated and refined. All lines of the powder photograph were satisfactorily indexed (Table 1) on the basis of cell edges which, when rounded off, are $a = 8.23$, $b = 12.88$, and $c = 6.26$ Å.

Powder photographs of natural lecontite may be indexed on a unit cell with edges within 0.03 Å of that used to index synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$. Moreover, the powder patterns of these two materials are remarkably similar (Table 2), except that natural lecontite possesses several extra lines which are attributable to small admixtures of other minerals as will soon be discussed.

For both natural lecontite and the synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$, the major lines reported in the literature around $d = 5.07$ and $d = 3.85$ – 3.87

TABLE 1. INDEXED POWDER DATA FOR ARTIFICIAL $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}^1$

Observed Values					Calculated Values ²			
hkl	I	d(Å)	2θ	Q	d(Å)	2θ	Q	ΔQ×10 ⁶
020	22	6.440	13.75	0.02411	6.439	13.75	0.02412	1
011	39	5.626	15.75	.03159	5.630	15.74	.03155	4
120	100	5.068	17.50	.03894	5.070	17.49	.03890	4
101	17	4.983	17.80	.04028	4.981	17.81	.04030	-2
111	61	4.646	19.10	.04632	4.646	19.10	.04633	-1
021	44	4.484	19.80	.04974	4.488	19.78	.04964	10
121	50	3.935	22.60	.06461	3.942	22.57	.06442	19
130	94	3.802	23.40	.06920	3.807	23.38	.06905	15
031	28	3.542	25.15	.07976	3.541	25.15	.07979	-3
201	61	3.445	25.87	.08432	3.439	25.91	.08464	-32
211	67	3.321	26.85	.09071	3.322	26.84	.09067	4
040	28	3.220	27.71	.09649	3.220	27.71	.09648	1
002	11	3.130	28.52	.10210	3.130	28.52	.10208	2
221	89	3.033	29.45	.10872	3.032	29.46	.10876	-4
140	11	3.003	29.75	.11089	2.998	29.80	.11126	-37
102	17	2.926	30.55	.11679	2.925	30.56	.11686	-7
041	28	2.863	31.25	.12207	2.863	31.24	.12200	7
231	78	2.686	33.35	.13855	2.683	33.40	.13891	-41
032	17	2.532	35.45	.15597	2.529	35.49	.15635	-38
212	11	2.445	36.75	.16722	2.445	36.75	.16723	-1
241	22	2.350	38.30	.18108	2.350	38.30	.18112	-4
330	33	2.313	38.94	.18695	2.311	38.98	.18729	-34
151	11	2.287	39.40	.19121	2.288	39.38	.19105	16
232	6	2.156	41.90	.21513	2.154	41.93	.21547	-34
013	17	2.060	43.95	.23563	2.060	43.96	.23571	-8
410	17	2.032	44.60	.24230	2.031	44.62	.24251	-21
023	22	1.985	45.70	.25374	1.985	45.71	.25380	-6
322	39	1.965	46.20	.25902	1.964	46.22	.25922	-20
123	11	1.929	47.10	.26863	1.930	47.10	.26858	5
033	6	1.877	48.50	.28386	1.876	48.51	.28395	-9
332	22	1.859	49.00	.28938	1.859	49.00	.28937	1
260	6	1.821	50.10	.30167	1.821	50.10	.30172	-5
351	6	1.797	50.80	.30960	1.798	50.77	.30929	31
440	6	1.734	52.80	.33268	1.733	52.82	.33296	-28
412	11	1.704	53.78	.34421	1.704	53.81	.34459	-38
441	6	1.671	54.96	.35830	1.670	54.98	.35848	-18
422	6	1.661	55.25	.36239	1.661	55.32	.36268	-29
262	6	1.626	56.60	.37821	1.626	56.61	.37828	-7
323	11	1.608	57.30	.38683	1.608	57.30	.38682	1
370	6	1.528	60.60	.42834	1.528	60.61	.42849	-15
512	6	1.447	64.40	.47782	1.447	64.38	.47761	21
044	11	1.408	66.39	0.50439	1.407	66.42	0.50480	-41

¹ The values of 2θ are for $\text{CuK}\alpha$ radiation.

² Based on direct and reciprocal constants: $a=8.23$, $b=12.88$, $c=6.26$; $a^*2=0.01478$, $b^*2=0.00603$, $c^*2=0.02552$.

were resolved into two lines if very thin powder rods were used. Use of such thin powder rods revealed that the strong line at $d = 5.07$ had overlapped a weaker line at $d = 4.98$; similarly the broad line in the region $d = 3.85-3.87$ was resolved into two lines, one in the region $d = 3.91-3.94$ and the second in the region $d = 3.78-3.81$.

Following the resolution of such lines by the use of ultra-thin rods, all lines of consequence could be indexed on the powder photograph of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$; whereas, in the powder photographs of natural lecontite, several lines remained unindexed. These lines are tentatively ascribed in Table 2 to thenardite Na_2SO_4 , mascagnite $(\text{NH}_4)_2\text{SO}_4$, and/or arcanite K_2SO_4 . The evidence for the presence of thenardite is quite strong. Four of its five most intense lines are present as unindexed lines in the lecontite patterns; whereas the fifth line ($d = 4.66$) coincides with an intense line of lecontite. Recognition of mascagnite and/or arcanite is difficult because several of the intense lines of these two minerals coincide with lines of lecontite. Mascagnite is probably present in the two Yale specimens. However, the U. S. National Museum specimen may either possess arcanite as an additional mineral or else the lines ascribed to mascagnite and arcanite are due to a member of a mascagnite-arcanite solid solution series, perhaps an ammonia-rich taylorite $(\text{K}_{2-x}(\text{NH}_4)_x\text{SO}_4$, in which x is approximately 0.33).

Computation of unit cells. The unit cells for the three lecontite samples were computed from the Q values of eighteen reflections whose indexing appeared unambiguous. These reflections were divided into three groups as follows: (1) 221, 410, 201, 212, 211, 231; (2) 020, 031, 032, 140, 021, 120; and (3) 101, 011, 111, 023, 013, 123. For each group of reflections, there was computed

$$\sum h^2a^{*2} + \sum k^2b^{*2} + \sum l^2c^{*2} = \sum Q_{hkl} \quad (1)$$

This done for each group, there was obtained a set of three equations in three unknowns, thus

$$36a^{*2} + 16b^{*2} + 8c^{*2} = Q_{221} + Q_{410} + Q_{201} + Q_{212} + Q_{211} + Q_{231} \quad (2)$$

$$2a^{*2} + 46b^{*2} + 6c^{*2} = Q_{020} + Q_{031} + Q_{032} + Q_{140} + Q_{021} + Q_{120} \quad (3)$$

$$3a^{*2} + 11b^{*2} + 30c^{*2} = Q_{101} + Q_{011} + Q_{111} + Q_{023} + Q_{013} + Q_{123} \quad (4)$$

Solution of these equations for the lecontite samples, using Q values obtained from the d -spacings cited in Table 2, yielded the following unit cells (values ± 0.03)

	a	b	c
Lecontite U.S.N.M. R6085	8.24	12.85	6.24 Å
Lecontite Yale Coll. 1696	8.23	12.86	6.25 Å
Lecontite Yale Coll. 4863	8.24	12.88	6.24 Å

TABLE 2. POWDER PATTERNS FOR ARTIFICIAL MATERIAL AND LECONTITE

hkl	Artificial Material				U.S.N.M.		Yale Collection			
	Author's		A.S.T.M. card 2-0161		R6085		1696		4863	
	d	I	d	I	d	I	d	I	d	I
020	6.439	22	6.50	60	6.440	12	6.440	20	6.436	17
011	5.630	39	5.65	70	5.634	37	5.623	40	5.634	33
120	5.070	100	5.07	100	5.062	75	5.085	81	5.085	83
101	4.981	17	—	—	4.975	50	4.980	60	4.972	67
111	4.646	61	4.64	80	4.644	100	4.661	100	4.649	83
021	4.488	44	4.48	60	4.488	32	4.491	40	4.486	33
Mascagnite?					4.338	57B	4.345	60	4.349	17
Arcanite or Taylorite?					4.183	32	—	—	—	—
121	3.942	50	3.85	80B	3.911	50	3.918	30	3.923	50
130	3.807	94	—	—	3.804	50	3.802	40	3.783	56
031	3.541	28	—	—	3.532	25	3.537	20	3.539	17
220	—	—	3.48	70	—	—	—	—	—	—
201	3.439	61	—	—	3.443	37	3.444	20	3.445	50
211	3.322	67	3.29	70	3.323	44	3.321	30	3.325	56
040	3.220	28	3.20	50	—	—	—	—	3.218	33B
Thenardite					3.189	32	3.191	20	—	—
002	3.130	11	—	—	3.134	19	3.137	20	—	—
Thenardite					3.075	25	3.078	30	—	—
221	3.032	89	3.03	80	3.035	89	3.037	81	3.038	100
140	2.998	11	—	—	2.995	25	2.994	30	2.998	10
102	2.925	17	—	—	—	—	—	—	2.931	10
041	2.863	28	2.87	60B	2.894	44B	2.868	30B	2.862	33
Thenardite					2.788	114	2.789	120	2.786	42
231	2.683	78	2.67	70	2.680	37	2.683	40	2.685	33
Thenardite					2.651	37	2.650	60	2.654	33

(Continued on facing page)

TABLE 2.—(Continued)

hkl	Artificial Material				U.S.N.M.		Yale Collection			
	Author's		A.S.T.M. card 2-0161		R6085		1696		4863	
	d	I	d	I	d	I	d	I	d	I
032	2.529	17	2.52	50	2.527	25	2.527	20	2.529	25
212	2.445	11	2.43	50	2.448	12	2.449	10	2.449	10
132	—	—	—	—	2.416	12	2.411	10	2.417	10
241	2.350	22	—	—	—	—	—	—	2.350	17
330	2.311	33	2.33	50	2.331	37B	2.328	40B	2.318	25B
151	2.288	11	—	—	—	—	—	—	—	—
—	—	—	2.23	20	—	—	—	—	—	—
331	—	—	—	—	2.171	29	—	—	—	—
232	2.154	6	2.15	60	2.154	29	2.161	20B	2.155	25B
340	—	—	—	—	2.091	32	—	—	—	—
013	2.060	17	—	—	2.059	19	2.067	20B	2.062	17
410	2.031	17	2.04	50B	2.036	32	2.031	10	2.031	17
023	1.985	22	—	—	1.985	64	1.986	30	1.985	50
322	1.964	39	1.97	80	—	—	1.964	10	1.965	10
123	1.930	11	1.91	20	1.929	12	1.927	10B	1.926	10
260	—	—	—	—	1.900	12	—	—	—	—
033	1.876	6	—	—	—	—	—	—	—	—
332	1.859	22	1.86	60	1.868	12	1.868	51	1.869	33
Thenardite					1.846	12	—	—	—	—
261	1.821	6	—	—	—	—	—	—	—	—
351	1.798	6	1.79	20	1.807	12	1.798	10	—	—
440	1.733	6	1.73	60	1.735	12	1.734	10	1.735	17
412	1.704	11	1.70	20	—	—	—	—	—	—
441	1.670	6	—	—	1.682	19	1.682	20	1.684	10
422	1.661	6	1.66	40	1.665	19	1.663	20	1.663	10
262	1.626	6	—	—	—	—	—	—	1.626	10
323	1.608	11	1.61	50B	1.607	19	1.607	20	1.608	17
450	—	—	1.55	40	1.556	12	1.554	10	—	—
370	1.528	6	1.52	40	—	—	—	—	—	—
280	—	—	1.49	20	1.500	12	1.496	10	1.496	10
512	1.447	6	1.44	40	—	—	—	—	—	—
044	1.407	11	1.40	30	—	—	—	—	—	—
372	1.373	6	—	—	—	—	—	—	—	—
620	1.341	6	—	—	1.345	12	1.339	10	1.341	17
334	—	—	—	—	1.296	12	1.296	10	1.299	10
192	—	—	—	—	1.286	12	1.280	10	—	—

which compare very closely with the unit cell upon which the powder data for synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ was indexed ($a=8.23$, $b=12.88$, $c=6.26 \text{ \AA}$).

Previous orientations of lecontite. The axes labelled a , b , and c when Donnay's (1943) convention was applied to the foregoing unit cell edges were differently labelled by J. D. Dana (as cited in Palache *et al.*, 1951, p. 438) and by Winchell and Winchell (1951, p. 170). The a -, b -, and c -axes determined by the x-ray results equal, respectively, the b -, c -, and a -axes of J. D. Dana and the c -, b -, and a -axes of Winchell and Winchell. The transformation matrices are:

$$\begin{array}{cccc}
 \text{Dana to X-ray} & \text{X-ray to Dana} & \text{Winchell and} & \text{X-ray to Winchell} \\
 & & \text{Winchell to X-ray} & \text{and Winchell} \\
 \begin{bmatrix} 0 & 1 & 0 \\ 0 & 0 & 1 \\ 1 & 0 & 0 \end{bmatrix} & \begin{bmatrix} 0 & 0 & 1 \\ 1 & 0 & 0 \\ 0 & 1 & 0 \end{bmatrix} & \begin{bmatrix} 0 & 0 & 1 \\ 0 & 1 & 0 \\ 1 & 0 & 0 \end{bmatrix} & \begin{bmatrix} 0 & 0 & 1 \\ 0 & 1 & 0 \\ 1 & 0 & 0 \end{bmatrix}
 \end{array}$$

The axial ratios, computed from the edges of the unit cells, compare fairly closely with those cited in the literature (Table 3). To facilitate

TABLE 3. COMPARISON OF AXIAL RATIOS (a:b:c)

Identity of specimen	x-ray Orientation	J. D. Dana Orientation	Winchell & Winchell Orientation
Materials Studied			
$\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$	0.639:1.000:0.486	0.761:1.000:1.565	0.486:1.000:0.639
Lecontite U.S.N.M. R6085	0.641:1.000:0.486	0.757:1.000:1.559	0.486:1.000:0.641
Lecontite Yale Coll. 1696	0.640:1.000:0.486	0.759:1.000:1.562	0.486:1.000:0.640
Lecontite Yale Coll. 4863	0.640:1.000:0.484	0.757:1.000:1.563	0.484:1.000:0.640
Values from Literature			
Lecontite (Palache <i>et al.</i> 1951, p. 438)	0.653:1.000:0.512 ³	0.7848:1.000:1.5317 ¹	—
Lecontite (Winchell and Winchell, 1951, p. 170)	0.633:1.000:0.486	—	0.486:1.000:0.633 ²

¹ Axial ratio cited by Palache *et al.* (1951, p. 438) for natural lecontite.

² Axial ratio cited by Winchell and Winchell (1951, p. 170) for natural lecontite.

³ Calculated from literature data.

comparison, the axial ratios for the materials here studied were also computed for the Dana orientation and the Winchell and Winchell orientation. Conversely, the axial ratio reported by Dana and that reported by Winchell and Winchell were recomputed for the x -ray orientation.

Physical properties. The optical properties of synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ crystals are: $\alpha = 1.440 \pm 0.002$, $\beta = 1.454 \pm 0.002$, $\gamma = 1.455 \pm 0.002$; $(-)$, $2V = 29^\circ 44'$ (calc.)¹; $X = c$, $Y = a$, $Z = b$. These compare closely with comparable data cited by Palache *et al.* (1951, p. 438) for lecontite, namely: $\alpha = 1.440 \pm 0.003$, $\beta = 1.452 \pm 0.003$, $\gamma = 1.453 \pm 0.003$; $(-)$, $2V = 40^\circ \pm 1^\circ$ (meas.).

On the basis of a unit cell content of $Z = 4$, the density of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ was calculated as 1.737. This compares fairly well with 1.745, the density value measured by the sink-float method in an α -monochloronaphthalene-diiodomethane solution using the curves of Bloss (1961, p. 64).

Using the axial setting $c < a < b$ (x -ray orientation), the crystals of $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ were short prismatic, being slightly elongated in the c -axis direction. Also using this setting the crystals exhibited a $\{011\}$ cleavage.

DISCUSSION OF RESULTS

Based on the close agreement of the physical properties, optical properties and diffraction data, it is believed that synthetic $\text{NaNH}_4\text{SO}_4 \cdot 2\text{H}_2\text{O}$ is identical to lecontite. This being the case, the crystal class of lecontite should be orthorhombic disphenoidal-222 instead of the currently accepted orthorhombic dipyramidal- $2/m \ 2/m \ 2/m$.

It is also believed that a few lines in lecontite powder patterns are due to admixtures of thenardite, mascagnite and/or arcanite. If this is true and was previously unrecognized, the chemical analyses of lecontite may be in error as to potassium, ammonium and/or sodium.

The density, which has not been reported for lecontite, was measured as 1.745 for the synthetic crystals and calculated to be 1.737 on the basis of $Z = 4$.

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¹ The calculated value for $2V$, $29^\circ 44'$ from our refractive indices, differs appreciably from the measured 40° $2V$ in Palache *et al.* (1951, p. 438). The $2V$ calculated from the refractive indices cited in Palache *et al.* is $31^\circ 56'$, not far from that of the writers'.

The accuracy of calculated $2V$ depends upon the accuracy by which the indices of refraction were determined. Although our determinations are reported ± 0.002 , we are certain that $\gamma - \beta$ does not exceed 0.002. If α was measured 0.002 too high or low then $2V$ would be about 2° smaller or larger. Hence the precision of the calculated $2V$ is approximately $\pm 3^\circ$.

samples of the rare mineral lecontite. Drs. Stanely E. Harris, Jr. and Dewey H. Amos of Southern Illinois University kindly read the manuscript. The Graduate Council of Southern Illinois University and, in particular, Dr. John Anderson were instrumental in obtaining funds to purchase the x-ray equipment which made this study possible.

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