

this study). Although stronger single crystal patterns may show additional weak reflections, it appears unlikely that the 002 and 004 reflections are correctly indexed on the powder pattern, since they would never become stronger than the 003 reflection, present in the precession photograph, but not observed in the powder photograph. The intensity of the 00 $l$  reflections in the powder photograph may be weakened by preferred orientation of the fibers, parallel or at small angles to the spindle axis. The very weak 8.00 and 4.02 Å reflections on the stronger powder photograph may correspond to possible (110) and (200) reflections, missing on the weaker precession photographs, but the 3.173 Å reflection cannot be indexed as an FeK $\alpha$  reflection. All three reflections, as well as other reflections known to be beta reflections, are enhanced in white radiation; therefore, the d-spacings of the three reflections have been recalculated from the measured  $2\theta$  using  $\lambda$ , FeK $\beta$  = 1.7568 (Table 2).

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## LITHIAN HUREAULITE FROM THE BLACK HILLS

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In 1946 the writer published an abstract describing what was thought to be a new pegmatite "phosphate of lithium, with iron and manganese" which was given the name *bastinite*. On the basis of work with the Universal stage it was decided the mineral was triclinic. At this time the mineral had not been examined with  $x$ -rays. Weissenberg pictures taken late in 1947 indicated that the mineral was monoclinic and powder pictures (Table 1) taken a few months later confirmed that it was a variety of hureaulite. At this time I was too busy with other things to finish the study of this mineral, and unfortunately the name got preserved by Chudoba (in Hintze) and in Strunz's tables, although later communications to these gentlemen corrected the matter.

A few samples of hureaulite were discovered in 1825 by the ceramist Alluaud in the pegmatite quarry of Huréaux at St. Sylvestre, according to LaCroix (1962). After the original discovery no more samples were found here. LaCroix states that from 1849-52 samples now in museum collec-

TABLE 1. DATA FROM POWDER PHOTOS OF HUREAULITE<sup>1</sup>

(h k l) <sup>2</sup>	Computed <sup>3</sup> d	Bauldaufite (Strunz)		Wenzelite (Strunz)		Wenzelite P37		La Vilate (Strunz)		Limooges P88		Palmita P49		Branchville P90		Black Hills P46	
		Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d
200	8.71	m	8.415	m	8.415	6	8.690	m	8.786	6	8.623	7	8.758	6	8.690	4	8.734
110	8.08	m	7.777	m	7.729	8	8.063	m	7.977	7	8.005	9	8.063	7	8.005	5	8.005
111	6.325					2	6.332			2	6.296	3	6.296	3	6.261	4	6.368
111	6.00					2	5.994			2	5.946	3	5.994	3	5.962	1	5.931
(310)	4.87					4	4.901			1	4.943	1	4.943	1	4.901		
002	4.75	s	4.633	s	4.610	2	4.717	s	4.674	2	4.678	3	4.717	3	4.698	1	4.698
311	4.48	s	4.468	s	4.465	3	4.529	s	4.489	4	4.502	4	4.547	5	4.511	2	4.511
311,400	4.35-18					1	4.372			2	4.347	4	4.339	2	4.355	4	4.339
021,220	4.08-.04	s	4.002	s	4.025	2	4.061	s	4.031	3	4.054	3	4.046	3	4.054	1	4.061
221	3.76	ss	3.718	ss	3.715	1	3.792	ss	3.780	2	3.773	3	3.792	2	3.767	4	3.767
221	3.64	ss	3.592	ss	3.599	2	3.648	ss	3.602	3	3.636	4	3.648	3	3.636	1	3.625
312	3.57											4	3.558			4	3.493
402	3.41		nicht messbar			1	3.351	sss	3.382	1	3.400	1	3.400	1	3.390	4	3.391
022,312	3.29-.25					3	3.257			4	3.253	5	3.257	5	3.261	3	3.257
511	3.19									6	3.177			6	3.186	5	3.186
222	3.16			st	3.126	10	3.152	st	3.128	10	3.135	10	3.143	10	3.135	10	3.139
421,402	3.07-.04									3	3.061	5	3.053	2	3.069	2	3.053
222	3.00	s	3.010	s	3.006	2	3.069	s	3.015	3	3.013			3	2.983	2	3.021
511	2.98	m	2.950	m	2.954	7	2.990	m	2.970	7	2.983	7	2.983	8	2.983	7	2.975
600	2.90									3	2.902	2	2.916	2	2.902	2	2.902

<sup>1</sup> All photos taken with filtered iron radiation, the Chicago ones with Straumanns (Philips) 114 mm. diameter cameras. The Chicago pictures were measured ( $\lambda=1.9373 \text{ \AA}$ ) by Leon Atlas in 1948-49. P37 is Harvard 81242, P88 is Yale (Brush 3159), presumably from "La Vilate." P49 from the Stewart Mine (Pala, Calif.) is from W. T. Schaller. P90 is Yale (Brush 3102). P46 is from the Custer Mt. Lode, SE of Custer, South Dakota.

<sup>2</sup> All indices represent planes confirmed from the study of single crystal (precession and Weissenberg) photos. Those given in parentheses furnished weak reflections, the others medium to strong ones.

<sup>3</sup> Spacings to (511) of  $d=3.19$  mostly computed graphically in 1949 by F. D. Bloss (*Am. Mineral.* 37, 588-599, 1952) using  $17.42:9.12:9.50$  with  $\beta$  of  $96^{\circ}40'$ .

TABLE 1—(continued)

(h k l) <sup>2</sup>	Computed <sup>3</sup> d	Bauldaufite (Strunz) Int. d	Wenzelite (Strunz) Int. d	Wenzelite P37 Int. d	La Vilate (Strunz) Int. d	Limoges P88 Int. d	Falate P49 Int. d	Branchville P90 Int. d	Black Hills P46 Int. d
113	2.896	sss 2.851	sss 2.834	4 2.880	sss 2.862	5 2.860	5 2.866	5 2.874	4 2.860
512	2.83						1 2.805	1 2.832	
313	2.79					1 2.766	1 2.766	1 2.766	
422	2.73	sss 2.720	sss 2.716		sss 2.712	1 2.722	1 2.722	1 2.728	‡ 2.728
330	2.69					1 2.691	1 2.691	1 2.691	‡ 2.691
331, 023, 602	2.65--.60	s-m 2.591	s-m 2.588	4 2.631	s-m 2.605	5 2.620	6 2.626	6 2.626	5 2.620
132, 512	2.56			2 2.564	s 2.529	1 2.580	2 2.586	2 2.586	1 2.586
331,	2.55	s 2.526	s 2.523			4 2.553	6 2.547	4 2.553	4 2.553
132	2.51						1 2.489		
221, 223	2.43			2 2.439		4 2.429	4 2.429	4 2.429	3 2.429
513, 711, 004	2.40--.38	s 2.397	s 2.408	2 2.400	s 2.403	3 2.391	4 2.396	4 2.391	3 2.391
602	2.35			1 2.358		2 2.354	1 2.358	2 2.354	1 2.363
621, 204	2.34--.32	sss 2.328	sss 2.312	1 2.322	sss 2.321	2 2.318	2 2.322	3 2.322	2 2.318
530	2.29					1 2.288	3 2.292	2 2.288	2 2.288
711	2.27					3 2.267	3 2.271	3 2.266	2 2.267
622, 114, 712	2.25--.24	ss 2.252	ss 2.249		ss 2.244	4 2.242	4 2.242	‡ 2.242	‡ 2.242
800, 133, 404	2.20--.18	ss 2.169	s-m 2.167	6 2.186	s-m 2.170	6 2.186	6 2.186	6 2.182	6 2.186
423	2.145	ss 2.132	ss 2.126	1 2.141	ss 2.129	2 2.141	3 2.148	3 2.141	2 2.145
133, 333	2.11							1 2.126	
622, 224	2.09--.08	sss 2.072	sss 2.067	1 2.074	sss 2.077	1 2.098	2 2.098	2 2.098	1 2.098
314	2.06							1 2.080	
623, 042,	2.04	ss 2.033	ss 2.034	1 2.050	ss 2.036	3 2.050	3 2.050	3 2.050	2 2.050
333, 440	2.02--.00			1 2.024		3 2.024	3 2.024	3 2.024	2 2.024
424, 404	1.99--.96	s 1.964	s 1.962	1 1.979	s 1.970	2 1.979	2 1.982	2 1.979	2 1.982

TABLE 1—(continued)

(h k l)*	Computed <sup>a</sup> d	Baldaufite (Strunz)		Wenzelite (Strunz)		Wenzelite P37		La Vilate (Strunz)		Limoges P88		Palaité P49		Branchville P90		Black Hills P46	
		Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d	Int.	d
730, 820	1.94-.93	ss	1.920	ss	1.915	1	1.931	ss	1.917	3	1.926	3	1.926	3	1.926	2	1.926
821, 802, 115	1.88-.87			‡	1.894	2	1.894			2	1.894	1	1.897	1	1.897	‡	1.892
(134), (623)	1.85-.84	sss	1.852	sss	1.849	1	1.859	sss	1.846	2	1.856	2	1.859	2	1.859	1	1.859
(115)	1.828			1	1.828	1	1.828			1	1.833	2	1.833	1	1.833	‡	1.833
424	1.814	sss	1.809	sss	1.806			sss	1.811	1	1.818	2	1.820	1	1.818	‡	1.818
151	1.786	sss	1.772	sss	1.770	1	1.788	sss	1.777	2	1.788	1	1.786	2	1.788	1	1.788
225	1.756									4	1.755					2	1.757
604, 822, 10-0-0, 350	1.74-.73	s	1.738	s	1.736	2	1.746	s	1.733	4	1.741	4	1.737	3	1.744	2	1.741
152, 804	1.705	s	1.696	s	1.693	2	1.710	s	1.694	4	1.706	3	1.706	3	1.708	2	1.706
152	1.688									1	1.687	1	1.687	1	1.687		
044	1.645	m	1.628	m	1.629	5	1.640	m	1.629	6	1.640	6	1.638	6	1.640	6	1.640
135,	1.62	sss	1.604	sss	1.598	‡	1.610	sss	1.605	2	1.610	1	1.610	2	1.610	1	1.613
10-2-2, 206, 135	1.590									2	1.595	1	1.597	2	1.595	1	1.599
10-2-1, 10-0-2, 153, 425	1.58-.57	s-m	1.573	s-m	1.570	5	1.582	s-m	1.574	6	1.582	6	1.580	6	1.580	6	1.584
(153)	1.566									1	1.566	1	1.563	1	1.566		
206, 060, 116	1.54-.52	s	1.522	s	1.516			s	1.516			2	1.523	2	1.529		
061, 226	1.502					1	1.510					2	1.510	2	1.510		
10-2-2	1.49	s	1.487	s	1.485			s	1.485	2	1.494	3	1.493	3	1.494	2	1.495
226, 12-0-0	1.45	s	1.453	s	1.449	2	1.456	s	1.449	4	1.459	4	1.456	4	1.459	4	1.459
154	1.433	ss	1.431	ss	1.428	1	1.436	ss	1.429	2	1.436	2	1.436	2	1.436	1	1.437
136	1.411	ss	1.408	ss	1.406	1	1.419	ss	1.408	2	1.418	2	1.415	2	1.416	1	1.418
		s	1.382	s	1.379	1	1.388	s	1.382	3	1.391	3	1.388	3	1.391		

tions were taken from the pegmatite of La Vilate (just east of Route N20 close to Barost creek, and also known as Les eaux vertes) about one mile north of Chanteloube (the latter is 26 km north and a bit east of Limoges, and 7 km north of St. Sylvestre), but this deposit has now long been abandoned.

The results obtained from *x*-ray powder pictures are given in Table 1. The spacings and intensities of the stronger lines for hureaulite are thus 3.16 (10), 8.08 (7), 2.98 (7), 8.71 (6), 2.18 (6), 1.64 (6), 1.58 (6), 2.62 (5). The indexing in this table is based on the study of both precession and Weissenberg photographs made on samples from the Custer Mountain Lode in the Black Hills (Fisher, 1945) and from a crystal from the Stewart Mine, Pala, California, kindly sent me by Professor Joseph Murdoch. The results from the crystals from these two localities are in full agreement. Both J. D. McCullough (in Murdoch, 1943) and H. Strunz (1954), on the basis of study of rotation pictures, concluded the space group to be #13- $C_{2h}^4$ , although McCullough gave the orientation  $P2/c$ , whereas Strunz used  $P2/n$ . My pictures show the limitations on reflections to be  $hkl-h+k=2n$ ;  $h0l-h=2n$ ,  $l=2n$ ; and  $0k0-k=2n$ . In short the space group is either #9- $C_s^4-Cc$  or #15- $C_{2h}^6-C2/c$ , presumably the latter. The cell dimension obtained from precession photos of the Black Hills material are  $17.64 \text{ \AA} \pm .04$ ;  $9.13 \pm .02$ ;  $9.49 \pm .02$ ;  $\beta = 96^\circ 30' \pm .02'$ . As noted by Strunz the mineral becomes colored after bombardment by *x*-rays for a few hours; the Black Hills sample turned dark pink.

The results obtained from chemical analyses of hureaulite are shown in Table 2, which omits a number of apparently less-reliable efforts. The analysis of the Black Hills material was made on a sample of 40 mg. by Brynjolf Bruun for Fe, Mn and P. The determinations of Li, Ca and Mg were made spectroscopically by Oiva Joensuu. In the spring of 1948 Edward D. Goldberg ran a neutron-activation analysis for P on the Black Hills sample at the Argonne Laboratory. His results showed that 14.69 g of hureaulite carried 2.13 g of P; his standard was  $(\text{NH}_4)\text{H}_2\text{PO}_4$ . Goldberg thought his result (14.5% P, or 33.22%  $\text{P}_2\text{O}_5$ ) had a probable error of 10%, but apparently it was closer to 20%.

The optical properties that have been determined for hureaulites are summarized in Table 3. All are in agreement that the crystals are (-) and the orientation has  $\alpha \rightarrow [b]$ , with  $\gamma$  lying in (obtuse) angle  $\beta$ . My results on the Black Hills sample for the dispersion of  $\gamma$  in this position are not however in agreement with what is given in LaCroix. The value of  $\beta$  varies from 1.653 at  $\lambda$  of 6200 to 1.660 at  $\lambda$  of 5360. In general as is to be expected the Fe-rich members show higher *n*-values than the Fe-poor ones; however the results given for wenzelite in column (5) are an excep-

TABLE 2. CHEMICAL ANALYSES OF HUREAULITE

	(1)	(2)	(3)	(4)	(5)	(6)	(7)
Li <sub>2</sub> O				tr			2.1 ± 0.3
CaO				1.77	2.02	1.33	not > 0.7
MgO					0.26	0.40	not > 1.7
FeO		7.87	4.56	7.48	10.57	11.34	6.
Fe <sub>2</sub> O <sub>3</sub>				0.16			
MnO	48.66	41.66	42.29	40.87	36.01	36.16	37.
P <sub>2</sub> O <sub>5</sub>	38.98	38.00	38.36	39.02	38.83	38.91	43. ± 3.
H <sub>2</sub> O	12.36	11.98	12.20	10.43	12.42	12.37	n.d.
Insol.		0.38	2.70	0.89			
Total	100.00	99.89	100.11	100.62	100.11	100.51	
G	3.23	3.191	3.149	3.17 ± .03	3.196	3.194	3.21

(1) Computed for Mn<sub>3</sub>H<sub>2</sub>(PO<sub>4</sub>)<sub>4</sub>·4H<sub>2</sub>O.

(2) La Vilate, France. Average of three (Damour 1858)

(3) Branchville, Conn. Average of two (Wells 1890)

(4) Palaite (Schaller 1912). Note Murdoch found only 0.1% Fe.

(5) Baldaufite (Strunz 1954)

(6) Wenzelite (Strunz 1954)

(7) Black Hills (new)

tion. It also seems likely that the value given for  $\alpha$  in column (3) is a bit too high.

The morphology of hureaulite is described in some detail by Strunz (1954). Most of the Black Hills crystals are much like those of Fig. 1 of Strunz, except that  $\{\bar{2}01\}$  is so large that it tends to eliminate  $\{\bar{1}11\}$  or reduce it to a "line" face between (201) and the prism.

TABLE 3. OPTICAL PROPERTIES OF HUREAULITE

	(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)
$\alpha$	—	1.647	1.652	1.652	1.646	1.652	1.649	1.637
$\beta$	1.66	1.654	1.656	1.657	1.655	1.658	1.655	1.645
$\gamma$	—	1.660	1.660	1.662	1.657	1.663	1.659	1.649
2V	$\left\{ \begin{array}{l} 73\frac{1}{2} \text{ red} \\ 74 \text{ yel} \\ 75 \text{ red} \end{array} \right.$	74	—	—	—	85 (cpd)	$\left\{ \begin{array}{l} 76 \text{ red} \\ 77 \text{ green} \end{array} \right.$	$\left\{ \begin{array}{l} 76^{\circ}03'(\lambda=578) \\ 76^{\circ}14'(\lambda=546) \end{array} \right.$
$\gamma \wedge [C]$	$\left\{ \begin{array}{l} 75 \text{ red} \\ 76 \text{ blue} \end{array} \right.$	75	—	—	—	66	$\left\{ \begin{array}{l} 57 \text{ red} \\ 52 \text{ green} \end{array} \right.$	$\left\{ \begin{array}{l} 66 \text{ red} \\ 64 \text{ green} \end{array} \right.$
$\gamma - \alpha$	—	.013	.008	.010	.011	.011	.010	.0119

(1) La Vilate, France, LaCroix.

(2) Branchville, Conn. Schaller.

(3) Palaite. Schaller.

(4) Baldaufite.

(5) Wenzelite.

(6) Wenzelite. Strunz, 1954.

(7) Black Hills. Fisher.

(8) Mangualde, Portugal.

} Larsen and Berman.

A grain of Black Hills hureaulite held on a small loop of Pt wire above a candle flame fuses readily to a brown glass. The fusibility is closer to 1, rather than 3 as given in Winchell (1951). The hardness is very close to 4. The color varies from colorless to rose pink.

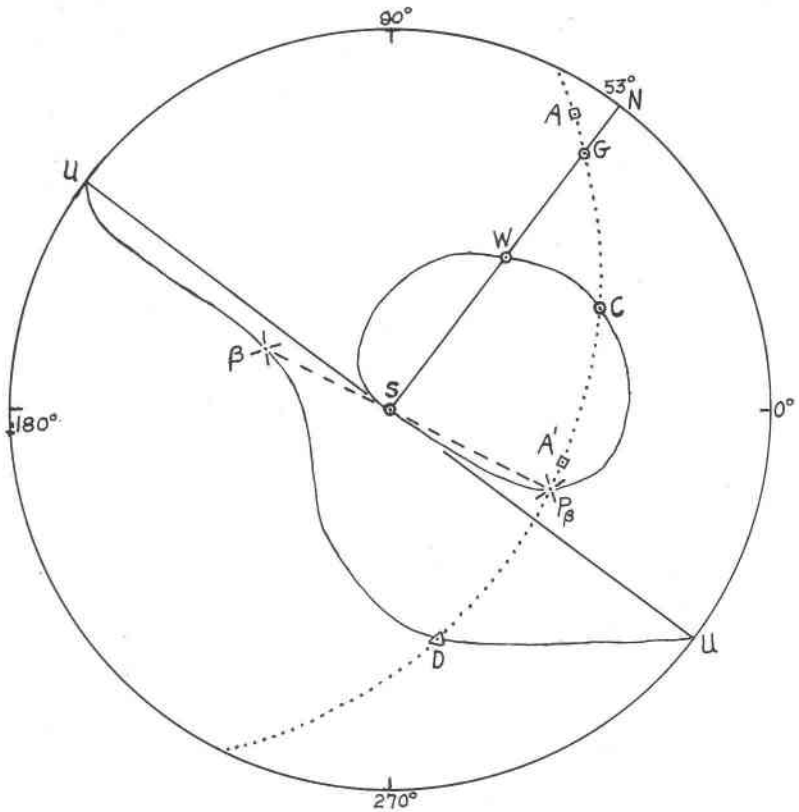


FIG. 1. Stereogram showing extinction curves given by the Mangualde hureaulite on the spindle stage.

Along the cracks (fracture surfaces) cutting the lithiophilite of the Custer Mt. Lode are found lilypad-shaped films of sicklerite or its alteration products; the hureaulite occurs as drusy aggregates fringing but not overlapping these, and is thought to be of very late hydrothermal origin. The paragenesis is discussed in detail in an earlier report (Fisher, 1945).

It is a pleasure to acknowledge the receipt of specimens used in this study from Professors Frondel, Murdoch and Winchell, and from Dr. Schaller.

## ADDENDUM

After the above paper had been completed, I received a hureaulite sample from Professor Joseph Murdoch of the University of California (Los Angeles) which he had collected from the Mesquitela pegmatite, Mangualde, Beira, Portugal in 1955. One crystal was mounted on the Umrig (Fisher, D. J., 1960, *Zeit. Krist.*, **113**, 77) where it was determined that the optical orientation was as stated earlier, with  $\gamma - \beta = .0042$  and  $\beta - \alpha = .0077$  and that other data were as given in rows 4 and 5 of column (8) of Table 3. Paul Moore and Paul Ribbe of this department are studying the crystal structure of hureaulite; their work should be completed shortly.

Another crystal was mounted on the spindle stage to determine the indices of refraction ( $\pm .002$ ) as given in Table 3. The extinction curves obtained using Na light appear in Fig. 1, with the principal indices located at C (on the polar curve), and D and  $\beta$  on the equatorial curve. Particular care was taken in determining the spindle readings of 53 and 233, when extinction occurred as the analyzer vibration direction was parallel to the spindle axis. This permitted the plotting of the diameter UU with high accuracy. The radius SN drawn normal to UU served to locate point G where it cuts the optic plane. Then the formula (20) of Garacochea and Wittke (*Acta Cryst.* **17**, 183-189, 1964) after locating  $P_\beta$  (the polar point of  $\beta$ , where a diameter through  $\beta$  cuts the optic plane):

$$\cos 2V_c = \frac{\sin(CP_\beta - CG)}{\sin(CP_\beta + CG)}$$

gave the result of  $75^\circ 19'$  (with  $CP_\beta = 45\frac{1}{4}^\circ$  and  $CG = 31^\circ$ ), which is quite satisfactory; the optic axes A, A' were then added to the figure, assuming  $C = \alpha$  to be the acute bisectrix.

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#### USE OF THE NAKAMURA PLATE IN UNIVERSAL STAGE ORIENTATIONS

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The Nakamura plate is a refined biquartz wedge and can be used to determine accurately the optical orientation with the universal stage. It consists of two halves of equal thickness, one a right-handed quartz, the other a left-handed quartz which rotate the plane of polarization in opposite directions. The Nakamura plate divides the field into two halves and if the polarizer and analyzer are exactly crossed and the crystal is at extinction, the two halves of the plate will be exactly equally illuminated. Its use is similar to that of the biquartz wedge and has been described by Wright (1908).

The Nakamura plate is designed to be used at the focal plane of the ocular, between the polarizer and the analyzer, and the ocular must be adapted so as to permit its insertion. The Wright slotted ocular enables one to focus on the accessory plate and on the normal orthoscopic image at the same time. The slotted ocular used by this author is an improved Wright slotted ocular designed by Hallimond and Taylor (1948) which contains a rotatable internal sliding analyzer above the accessory slot. With wide tube microscopes it is necessary to construct a special joint in order to place the slot at the normal focal plane of the instrument.

Extinction positions with the Nakamura plate are more easily determined than with the normal orthoscopic image. When the mineral is at extinction both halves of the Nakamura plate are exactly equally illuminated, and if the crystal is rotated from extinction, the intensity of illumination in the two halves of the plate is rendered unequal. Extinction positions can be determined to 0.10 degree (Hallimond, 1953).

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