

EZCURRITE,  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ : A RESTUDY\*

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## ABSTRACT

New high quality specimens of ezcurrite from the original locality, the Tincalayu mine, Salta, Argentina, form the basis for the present study.

Ezcurrite is triclinic,  $\bar{1}$ ; space group  $P\bar{1}$ ;  $a=8.598 \text{ \AA}$ ,  $b=9.570 \text{ \AA}$ ,  $c=6.576 \text{ \AA}$ ,  $\alpha=102^\circ 45'$ ,  $\beta=107^\circ 30'$ ,  $\gamma=71^\circ 31'$ ;  $\lambda=82^\circ 00'$ ,  $\mu=75^\circ 32'$ ,  $\nu=105^\circ 39'$ ;  $a:b:c=0.8984:1:0.6871$ ; cell volume  $484.7 \text{ \AA}^3$ ;  $Z=1$ ; cell content  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ . Colorless well-formed crystals of 0.5 mm maximum length show the forms:  $\{001\}$ ,  $\{010\}$ ,  $\{100\}$ ,  $\{110\}$ ,  $\{1\bar{1}0\}$ ,  $\{101\}$ ,  $\{10\bar{1}\}$ ,  $\{\bar{1}\bar{2}6\}$ . The strongest lines in the x-ray powder photograph are in  $\text{\AA}$ : 6.936 (100); 3.074 (38); 4.494 (29); 3.135 (19); 2.780 (19).

Ezcurrite is optically biaxial (-);  $\alpha=1.468$ ,  $\beta=1.507$ ,  $\gamma=1.529$  (Na light);  $2V=73\frac{1}{2}^\circ$ ;  $r>v$ ; orientation:  $X \phi=177^\circ \rho=86^\circ$ ;  $Y \phi=-90^\circ \rho=47^\circ$ ;  $Z \phi=82^\circ \rho=43^\circ$ . Cleavage:  $\{110\}$  excellent,  $\{010\}$  good,  $\{100\}$  fair,  $\{1\bar{1}0\}$  poor, yielding fragments elongated on  $[001]$  also  $\{\bar{1}\bar{2}6\}$  fair,  $\{101\}$  poor. Hardness  $3 \parallel c$ ,  $3\frac{1}{2} \perp c$ . Specific gravity 2.053 (meas), 2.049 (calc).

A chemical analysis gives:  $\text{B}_2\text{O}_3$  59.53,  $\text{Na}_2\text{O}$  19.84,  $\text{K}_2\text{O}$  0.18,  $\text{H}_2\text{O}$  20.45. Ezcurrite and Suhr's borate (artificial) are identical; the formula for both is:  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ . D.T.A. shows endothermic peaks at  $280^\circ\text{C}$  (loss of  $\text{H}_2\text{O}$ ),  $575^\circ\text{C}$  (loss of OH) and  $780^\circ\text{C}$  (fusion). An exothermic peak at  $680^\circ$  results from the formation of a crystalline phase. A different crystalline phase is produced on cooling of the fused sample.

## INTRODUCTION

Ezcurrite,  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ , was described as a new mineral by Muessig and Allen (1957) from the Tincalayu borax mine, Salta, Argentina. The specimens they described were not found in place but collected from the mine dumps. Since that time and until recently the mine has been closed and no new material was available. With the reopening of the mine several specimens from the new workings have come to our attention that are of such fine quality that a restudy of the mineral seemed appropriate. These permitted more detailed work particularly with single crystals. Moreover, using this good material we hoped to resolve the question of the chemical composition raised by Cipriani (1961).

## OCCURRENCE

The geology of the Tincalayu deposit and the surrounding area has been described by Catalano (1930, 1964) and in a more detailed report, by Muessig and Allen (1957). The suggested genesis, by these latter authors, is that boron was deposited as borax in a playa together with clastic sediments and halite probably during late Cenozoic time. After burial by siltstones and sandstones, folding and faulting occurred, and

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“the original borax was probably pushed upward into a plug-like mass.” During this process the borax recrystallized into an extremely fine-grained aggregate with subparallel texture.

This microcrystalline borax comprises the bulk of the deposit with which are associated minor amounts of other rarer borates. These include kernite, (Ahlfeld and Angelelli, 1948), rivadavite (Hurlbut and Aristarain, 1967), and macallisterite and ezcurrite. The ezcurrite used in the present study is in nodular aggregates of elongated crystals embedded in the fine grained borax. Individual crystals having maximum dimensions of  $7.5 \times 1.4 \times 1.2$  centimeters are arranged in a subparallel manner,

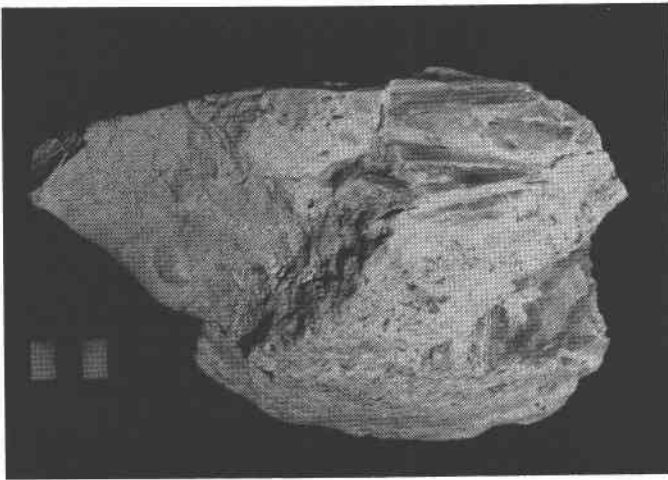


FIG. 1. Subparallel aggregates of ezcurrite crystals (upper and lower right) embedded in micro-crystalline borax superficially altered to tinalconite. Approximately one-third actual size; width of small rectangle represents one centimeter.

Figure 1. At the border of the nodules were found a few well-faced isolated crystals of ezcurrite, less than 0.5 millimeter in maximum dimension.

#### MORPHOLOGY

In the initial description, Muessig and Allen state, “The apparent lack of coincidence between optical direction and probable crystallographic axes suggest that ezcurrite is triclinic.” The present study confirms this suggestion.

The small crystals were used for a study of the morphology with the two-circle goniometer. The crystals are elongated on  $[001]$  and terminated by three major faces (Fig. 2). Several other small terminal faces were observed but with the exception of  $[\bar{1}26]$  are not recorded because of poor

quality and lack of signals. The angles and constants given in Table 1 are refined using X-ray measurements but the difference between measured and calculated values was in no case greater than 15 minutes. Figure 3 is a stereographic projection of the observed crystal forms and shows the adopted crystallographic orientation.

#### PHYSICAL AND OPTICAL PROPERTIES

The most characteristic feature of ezcurrite is the pronounced cleavages permitting the mineral to break into splintery fragments elongated parallel to  $[001]$ . There are four such cleavages:  $\{110\}$  excellent,  $\{010\}$

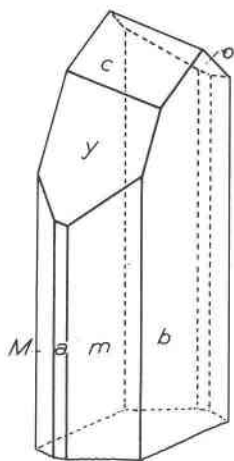


FIG. 2. Ezcurrite. Crystal drawing showing major forms.

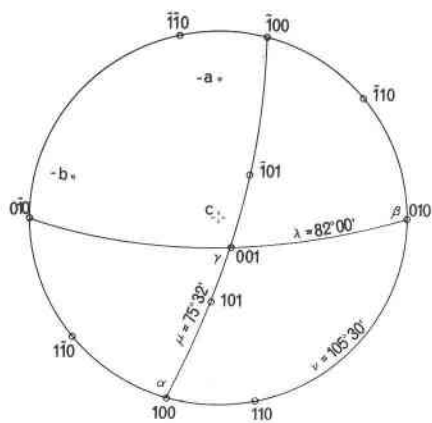


FIG. 3. Stereographic projection of the crystal forms shown in Figure 2, and the adopted crystallographic orientation.

good,  $\{100\}$  fair, and  $\{\bar{1}\bar{1}0\}$  poor. In addition there are two other cleavages:  $\{\bar{1}26\}$  fair and  $\{\bar{1}01\}$  poor. Muessig and Allen (1957) report good cleavages (probably in the  $[001]$  zone) intersecting at an angle of  $71^\circ 18.5'$  forming bent columnar fragments. This angle is impossible to reconcile with our measurements of  $(010) \wedge (110) = 57^\circ 42'$ ;  $(010) \wedge (100) = 105^\circ 39'$  ( $74^\circ 21'$ );  $(100) \wedge (110) = 47^\circ 57'$ . The only angles between cleavages approaching the value of  $71^\circ 18.5'$  are:  $(010) \wedge (\bar{1}01) = 71^\circ 31'$  and  $(\bar{1}00) \wedge (\bar{1}01) = 71^\circ 00'$ . In the specimens used in the present study we were unable to find the bent columns reported by Muessig and Allen.

The crystals of ezcurrite are colorless and transparent with a vitreous luster; a satiny appearance results from the intersection of the several cleavages in  $[001]$ , and is enhanced in some crystals by numerous, tiny

inclusions (rivadavite?) elongated parallel to the  $c$ -axis of ezcurrite. Although the specimens come from an area of very low humidity no alteration is observable after exposure for over a year to the high humidity of Cambridge, Massachusetts.

The hardness is 3 parallel to the length of the cleavage fragments, but distinctly greater, about  $3\frac{1}{2}$ , across the length. The average specific gravity as measured by suspension in a mixture of bromoform and acetone agreed exactly with the average of the determinations made on the Berman balance. The value obtained was 2.053, and checks closely with the calculated specific gravity, 2.049, determined for the theoretical chemical composition. The value given by Muessig and Allen (1957) was 2.153.

TABLE 1. EZCURRITE—ANGLE TABLE TRICLINIC; PINACOIDAL— $\bar{1}$ 

$$a:b:c=0.8984:1:0.6871; \alpha 102^{\circ}45'; \beta 107^{\circ}30'; \gamma 71^{\circ}31'$$

$$p_0:q_0:r_0=0.7865:0.6910:1; \lambda 82^{\circ}00'; \mu 75^{\circ}32'; \nu 105^{\circ}39'$$

$$p_0' 0.8330, q_0' 0.7318, x_0' 0.3159, y_0' 0.1473$$

Forms:	$\phi$	$\rho$	$A$	$B$	$C$
$c$ 001	65° 00'	19° 13'	75° 32'	82° 00'	0° 00'
$b$ 010	0 00	90 00	105 39	0 00	82 00
$a$ 100	105 39	90 00	0 00	105 39	75 32
$m$ 110	57 42	90 00	47 57	57 42	70 57
$M$ $\bar{1}\bar{1}0$	140 01	90 00	34 22	140 01	85 07
$y$ 101	93 58	48 15	43 04	92 58	32 28
$o$ $\bar{1}01$	-52 35	31 28	119 00	71 31	43 28
$r$ $\bar{1}\bar{2}6$	107 59	10 51	79 09	93 20	13 25

Tests for piezoelectricity and pyroelectricity gave negative results which suggest that the crystal class is pinacoidal.

The indices of refraction obtained in the present study agree well with those already published. The relation between the morphology and the optical indicatrix was determined. These results are summarized in Table 2 and shown graphically in Figures 4 and 5.

#### X-RAY STUDY

The unit-cell dimensions were determined from precession photographs with  $a$ ,  $b$  and  $c$  as precession axes (molybdenum radiation and zirconium filter). In addition cone axis and rotation photographs were made. The values given in Table 3 were refined using X-ray powder photographs. The space group is probably  $P1$  and is so given because tests for pyroelec-

TABLE 2. OPTICAL PROPERTIES OF EZCURRITE

	1	2
$\alpha$ (Na)	1.468	1.472
$\beta$ (Na)	1.507	1.506
$\gamma$ (Na)	1.529	1.526
	$\pm 0.001$	
	$\pm 0.002$	
Optic sign	Biaxial (-)	Biaxial (-)
$2V$	$73\frac{1}{2}^\circ$ (meas.)	$73\frac{1}{2}^\circ$ (cal.)
Dispersion	$r > v$	
Orientation:	$\phi$	$\gamma \wedge \text{elongation } 42.6^\circ$
	$X \ 177^\circ \ 86^\circ$	
	$Y \ -90^\circ \ 47^\circ$	
	$Z \ 82^\circ \ 43^\circ$	
	Face:	
Extinction angles	(010) $Z' \wedge c = 42\frac{1}{2}^\circ$	
observed on cleavage	(110) $Z' \wedge c = 3^\circ$	
faces in the [001]	(100) $Z' \wedge c = 12^\circ$	
zone	(1\bar{1}0) $Z' \wedge c = 27^\circ$	

1. This study.
2. Data from Muessig and Allen (1957).

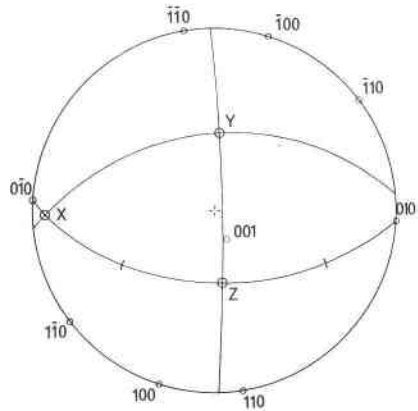
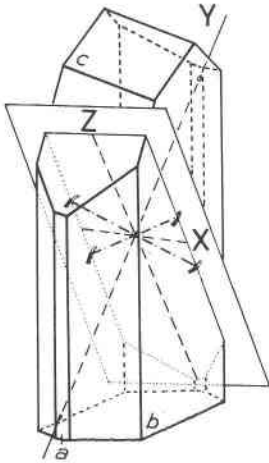


FIG. 4. Optical orientation of ezcurrite.

FIG. 5. Stereogram of ezcurrite showing optical orientation.

tricity and piezoelectricity were negative, and etch tests suggested a center of symmetry.

In Table 4 are given the spacings obtained from powder photographs (Cu/Ni) but with intensities derived from diffractometer charts. Calculated  $d$  values and indices were determined with an IBM 7094 computer. Also in Table 4 are the spacings and intensities given by Muessig and Allen in the original description of ezcurrite.

#### CHEMICAL COMPOSITION

In 1937, H. B. Suhr obtained a U. S. patent on a *New Borate Compound and Method of Preparing Same*. For this compound he proposed the formula,  $3\text{Na}_2\text{O} \cdot 8\text{B}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$ , that subsequently has been referred to in the literature as *Suhr's borate*. In 1957 Muessig and Allen described a new

TABLE 3. UNIT-CELL DATA FOR EZCURRITE

$a = 8.598 \text{ \AA}$ $b = 9.570 \text{ \AA}$ $c = 6.576 \text{ \AA}$	$\pm 0.002 \text{ \AA}$	Triclinic - $P\bar{1}$
$\alpha = 102^\circ 45'$ $\beta = 107^\circ 30'$ $\gamma = 71^\circ 31'$	$\pm 3'$	$\alpha^*(\lambda) = 82^\circ 00'$ $\beta^*(\mu) = 75^\circ 32'$ $\gamma^*(\nu) = 105^\circ 39'$
$a:b:c = 0.8984:1:0.6871$		Cell volume = $484.7 \text{ \AA}^3$ $Z = 1$

Specific gravity: meas. 2.053; calc. 2.049 for  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$

natural borate, ezcurrite, for which they proposed the formula  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$  (triclinic?).

In 1961 Cipriani and Vannunccini described two new borates from Larderello, Italy, one of which, named nasinite, is monoclinic with the proposed formula,  $2\text{Na}_2\text{O} \cdot 5\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ . Later Cipriani (1961), aware of both ezcurrite and Suhr's borate, determined that the two substances have similar optical properties and give the same X-ray powder pattern. He also made a chemical analysis of Suhr's borate (Table 5) from which he concluded that the original formula,  $3\text{Na}_2\text{O} \cdot 8\text{B}_2\text{O}_3 \cdot 10\text{H}_2\text{O}$ , was correct. Because of the differing formulas it was considered that ezcurrite and nasinite were not dimorphs.

In 1963, R. C. Erd reported that both he and N. P. Nies independently also established the identity between ezcurrite and Suhr's borate. He further stated that Nies analyzed Suhr's borate and reported the chemical formula as:  $2\text{Na}_2\text{O} \cdot 5.1\text{B}_2\text{O}_3 \cdot 7\text{H}_2\text{O}$ .

TABLE 4. X-RAY POWDER DATA FOR EZCURRITE

Present Study				Muessig and Allen (1957)		Present Study				Muessig and Allen (1957)	
<i>hkl</i>	<i>d</i> (obs) <sup>1</sup>	<i>d</i> (calc) <sup>2</sup>	<i>I</i> <sup>3</sup>	<i>d</i> (obs) <sup>4</sup>	<i>I</i>	<i>hkl</i>	<i>d</i> (obs) <sup>1</sup>	<i>d</i> (calc) <sup>2</sup>	<i>I</i> <sup>3</sup>	<i>d</i> (obs) <sup>4</sup>	<i>I</i>
010	8.966	8.989	6	8.98	1	4 $\bar{1}$ 1	1.665	1.664	<1	1.670	<1
—	7.668*	—	1	7.69*	1	0 $\bar{4}$ 3	1.636	1.640	2	1.642	<1
110	6.936	6.930	100	6.94	10	520	1.636	1.634			
$\bar{1}$ 01	5.614	5.609	<1	5.50	<1	332	1.616	1.618	<1		
$\bar{1}$ 10	5.274	5.270	1	5.28	<1	$\bar{3}$ 32	1.589	1.588	1	1.590	<1
011	4.781	4.808	1	4.82	<1	223	1.566	1.567	<1		
020	4.494	4.494	29	4.48	2	441	1.544	1.544	1	1.540	<1
$\bar{1}$ 11	4.200	4.199	3	4.20	<1	$\bar{3}$ 40	1.519	1.519	2		
210	4.030	4.038	1	4.04	<1	412	1.519	1.519			
$\bar{2}$ 01	3.800	3.787	1	3.80	<1	$\bar{3}$ $\bar{3}$ 2	1.476	1.476	2	1.480	<1
220	3.447	3.465	1			104	1.456	1.456	<1	1.459	<1
$\bar{2}$ 10	3.302	3.302	8	3.31	2	$\bar{1}$ $\bar{7}$ 4	1.456	1.456			
121	3.221	3.221	<1	3.22	<1	$\bar{3}$ $\bar{1}$ 3	1.429	1.429	<1		
				3.19	<1	0 $\bar{6}$ 2	1.429	1.429			
$\bar{2}$ 11	3.135	3.127	19	3.13	2	$\bar{0}$ $\bar{2}$ 2	1.415	1.419	<1	1.416	<1
$\bar{1}$ $\bar{3}$ 1	3.074	3.079	38	3.08	6	061	1.415	1.412	<1		
0 $\bar{1}$ 2		3.070				$\bar{4}$ 23	1.403	1.403	<1		
211	2.979	2.981	2	2.99	<1	$\bar{1}$ 63	1.374	1.374	<1	1.374	<1
0 $\bar{3}$ 1	2.854	2.858	<1	2.86	<1		1.357		<1		
$\bar{1}$ 12	2.780	2.781	19	2.77	3		1.333		<1		
022	2.739	2.739	<1				1.302		<1		
$\bar{3}$ 01	2.679	2.675	4	2.68	<1		1.270		<1		
221		2.678					1.256		<1		
300	2.626	2.632	2	2.64	<1		1.244		<1		
$\bar{1}$ 30	2.580	2.580	4	2.59	<1		1.214		<1	1.215	<1
131	2.526	2.535	2	2.53	<1		1.203		<1		
$\bar{3}$ $\bar{3}$ 1		2.441					1.183		<1		
$\bar{3}$ 22	2.432	2.418	<1	2.45	<1		1.158		<1		
$\bar{3}$ 10		2.360					1.137		<1		
$\bar{1}$ 41	2.362	2.360	8	2.36	1		1.126		<1		
140	2.333	2.333	2	2.32	<1		1.113		<1	1.112	<1
231	2.283	2.287	2	2.29	<1		1.097		<1		
040	2.241	2.247	<1	2.24	<1		1.086		<1		
202		2.190					1.076		<1		
$\bar{1}$ 13	2.192	2.190	1	2.21	<1		1.062		<1		
212	2.146	2.156	1	2.14	<1		1.043		<1		
$\bar{2}$ 23	2.104	2.105	2	2.10	<1		1.037		<1		
0 $\bar{1}$ 3	2.083	2.082	2	2.09	<1		1.026		<1		
$\bar{2}$ 31	2.039	2.035	10	2.04	<1		1.016		<1		
032		2.021					.996		<1		
420	2.020	2.019	8	2.02	<1		.978		<1		
$\bar{2}$ 31	1.990	1.990	2	1.994	<1		.967		<1		
$\bar{3}$ 13		1.958					.955		<1		
$\bar{1}$ 41	1.958	1.959	2	1.955	<1		.934		<1		
430	1.901	1.901	2	1.903	<1		.915		<1		
$\bar{3}$ 03	1.869	1.869	1	1.890	<1		.901		<1		
$\bar{4}$ 41	1.831	1.830	<1	1.802	<1		.889		<1		
$\bar{2}$ 52	1.791	1.793	3	1.793	<1		.863		<1		
$\bar{1}$ 52	1.764	1.764	<1				.847		<1		
$\bar{3}$ 52		1.733					.808		<1		
322	1.733	1.733	2	1.734	<1		.795		<1		
$\bar{2}$ 41		1.694									
151	1.693	1.694	<1	1.692	<1						

\* Rivadavite (?)

<sup>1</sup> Present study observed values obtained using: CuK $\alpha$ =1.54178 Å; CuK $\alpha$ =1.54051 Å; Ni filter; camera diameter 114.5 g mm.

<sup>2</sup> The calculated values were obtained on an IBM 7094 computer. Computer time was obtained on N.S.F. Grant No. GP-2372, Harvard University.

<sup>3</sup> The line intensities were derived from a diffractometer chart obtained with Cu radiation. For this purpose ezcurrite was ground with about 30 volume percent glass to minimize preferred orientation.

<sup>4</sup> Muessig and Allen's data obtained from a diffractometer chart using CuK $\alpha$ =1.5418 Å; Ni filter.

TABLE 5. CHEMICAL ANALYSES IN WEIGHT PERCENT OF EZCURRITE AND SUHR'S BORATE

	Ezcurrite				Suhr's borate		
	1	2	3	4	5	6	7
B <sub>2</sub> O <sub>3</sub>	59.53	58.86	58.99	58.20	59.83	60.35	58.68
Na <sub>2</sub> O	19.84	20.67	20.25	20.72	20.15	20.14	20.48
K <sub>2</sub> O	0.18	—	—	—	—	—	—
H <sub>2</sub> O	20.45	20.47	20.76	21.08	20.02	19.51	20.84
	100.00	100.00	100.00	100.00	100.00	100.00	100.00

1. Present Study. See Table 6.
2. Ralph Mathieson, *analyst*. Water by difference in Muessig and Allen (1957).
3. Henry Kramer, *analyst*. Recalculated to 100% in Muessig and Allen (1957).
4. Theoretical composition—ezcurrite, 2Na<sub>2</sub>O·5B<sub>2</sub>O<sub>3</sub>·7H<sub>2</sub>O.
5. C. Cipriani, *analyst*. Disregarding NaCl and recalculating to 100%. Cipriani (1961).
6. Theoretical composition—Suhr's borate, 3Na<sub>2</sub>O·8B<sub>2</sub>O<sub>3</sub>·10H<sub>2</sub>O.
7. Calculated from the formula, 2Na<sub>2</sub>O·5.1B<sub>2</sub>O<sub>3</sub>·7H<sub>2</sub>O, proposed by N. P. Nies, *analyst*, Erd (1963).

One of the goals of the present study was a clarification of the confusion surrounding the chemical composition of ezcurrite and Suhr's borate. A chemical analysis of ezcurrite made by Jun Ito is reported in column 3, Table 5. In the same table are given other analyses of ezcurrite and Suhr's borate. One will see by inspection of the table a close similarity between all the analyses.

TABLE 6. MOLECULAR PROPORTIONS OF EZCURRITE AND SUHR'S BORATE

Chemical analysis	Molecular proportions				
	1	2	3	4	5
	Wt. %	Wt. % recal.		Ezcurrite 1.13482=1	Suhr's borate 0.85484=1
B <sub>2</sub> O <sub>3</sub>	59.4	59.531	0.85484	0.75388×7=5.27	1×8=8
Na <sub>2</sub> O	19.8	19.844	0.32015	0.28381×7=1.99	0.37676×8=3.01
			0.32207		
K <sub>2</sub> O	0.18	0.180	0.00192	1×7=7	1.32752×8=10.62
H <sub>2</sub> O <sup>+</sup>	20.4	20.445	1.13482		
H <sub>2</sub> O <sup>-</sup>	0.0	—	—		
	99.78	100.00			

1. Ezcurrite. Jun Ito, *analyst*. H<sub>2</sub>O<sup>+</sup> between 140°-400°C.



Table 6 gives Ito's analysis and the molecular proportions obtained from it. The table also shows the molecular proportion using  $H_2O$  as unity (Column 4) and using  $B_2O_3$  as unity (Column 5). Multiplying these values by 7 and 8 respectively one obtains: essentially the molecular proportions given for the proposed ezcurrite formula,  $2Na_2O \cdot 5B_2O_3 \cdot 7H_2O$  (Column 4); and approximately the molecular proportions given for the formula of Suhr's borate,  $3Na_2O \cdot 8B_2O_3 \cdot 10H_2O$  (Column 5). Thus within narrow limits the two formulas express the same chemical composition, but with quite different molecular weights. The molecular weight of the first (ezcurrite) is 598.3 and of the second (Suhr's borate) is 923.2. However, from the unit cell dimension and specific gravity the calculated molecular weight is 599.4 with  $Z=1$ . This shows that the correct formula for ezcurrite and thus also for Suhr's borate is the one proposed by Muessig and Allen (1957). Consequently nasinite (Cipriani and Vannunccini, 1961) is a monoclinic dimorph of ezcurrite.

The formula that emerges from the chemical analysis made by wet methods for the present study is:  $1.99Na_2O \cdot 5.27B_2O_3 \cdot 7H_2O$ . The departure from the theoretical formula may be accounted for by inclusions, possibly of rivadavite,  $Na_6MgB_{24}O_{40} \cdot 22H_2O$ . This assumption seems reasonable because: 1) the strongest line of this mineral appears in the X-ray powder photograph (see Table 4). 2) a spectrochemical analysis shows a trace of magnesium 3) an unit volume of rivadavite has less  $Na_2O$  and  $B_2O_3$  but more  $H_2O$  than an unit volume of ezcurrite. However, from the analysis of the synthetic compound Nies (Erd, 1963) reports a formula with  $5.1B_2O_3$ .

Christ (1960) proposed four rules governing the nature of complex boron-oxygen polyions found in hydrated borates. Using these rules and other crystal-chemical arguments he proposed for ezcurrite the structural formula:  $Na_2[B_5O_6(OH)_5] \cdot H_2O$  resulting from a combination of three triangles and two tetrahedrons. The triangles are formed by boron linked to three oxygens and the tetrahedrons by boron linked to four oxygens. The triangles and tetrahedrons are joined by sharing oxygens, but the oxygens not shared attach a proton and exist as  $(OH)^-$ . An insular group so formed is a polyion,  $[B_5O_6(OH)_5]^{-2}$ . If this is correct, there will be two structural formulas per unit cell of ezcurrite, but only one formula weight.

#### DIFFERENTIAL THERMAL ANALYSIS

As pointed out by Muessig and Allen (1957) ezcurrite and kernite have similar physical appearance and can be easily confused by casual inspection. Because of the similarities, D.T.A. and X-ray diffractometer charts of the two minerals are reproduced in Figures 6 and 7.

A differential thermal analysis of ezcurrite shows a major endothermic

peak at 280°C. It is interpreted as resulting from the loss of structural water, following which the material becomes amorphous ( $n=1.510$ ). The broader endothermic peak at about 575° may result from the loss of (OH); and the final endothermic peak at 780° results from melting of the sample. The exothermic peak at 680°C is caused by the formation of a crystalline phase. The crystals are platy with the X optical direction perpendicular or nearly perpendicular to the plates. The optical properties are:  $\alpha=1.467$ ,  $\beta=1.504$ ,  $\gamma=1.531\pm 0.001$  (Na light); optically (-),

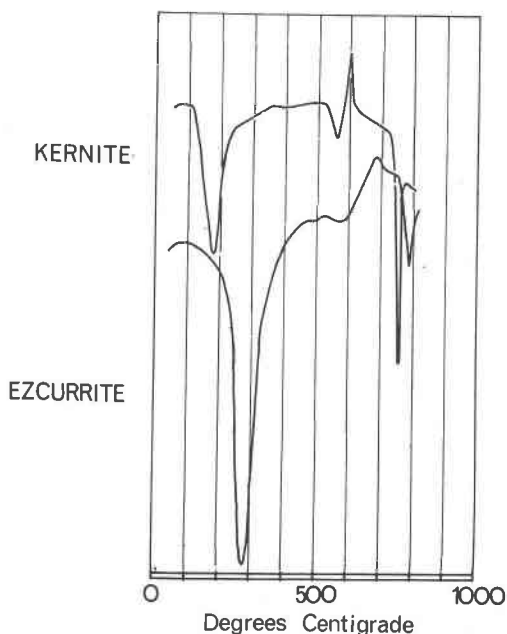


FIG. 6. D.T.A. curves of ezcurrite and kernite. Rate of heating 20°/mi; reference junction 0°C; thermocouple Pt/Pt+13% Rh; reference material Al<sub>2</sub>O<sub>3</sub>.

$2V=78^\circ$ . The spacings in Å and the relative intensities of the strongest peaks as obtained on an X-ray diffractometer chart are: 4.53 (100), 2.94 (55), 3.01 (50), 3.73 (30).

If the melt formed at 780° is quenched, a glass results with index of refraction of 1.512. However, if the melt is slowly cooled another crystalline phase is produced whose optical properties are:  $\alpha=1.458$ ,  $\beta=1.517$ ,  $\gamma=1.531$ ; optically (-),  $2V=55^\circ$ . The spacings in Å and the relative intensities of the strongest peaks as obtained on an X-ray diffractometer chart are: 4.46 (100), 1.73 (78), 8.78 (70), 2.56 (70), 2.98 (62), 3.46 (60), 3.34 (58).

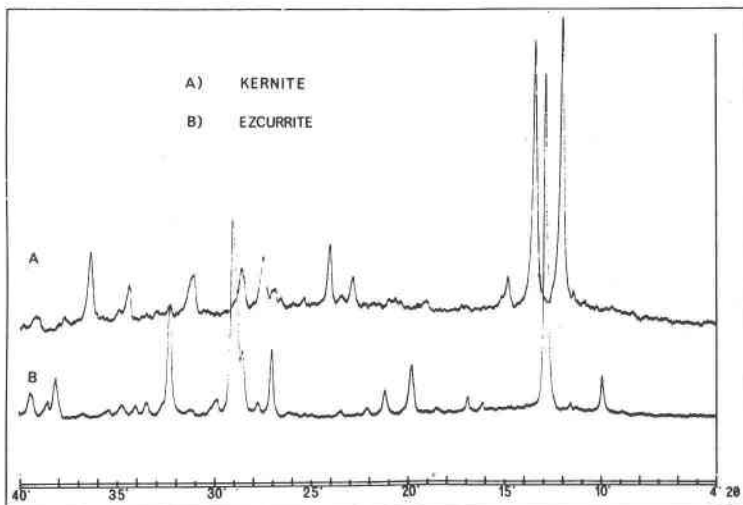


FIG. 7. Diffractometer charts of ezcurrite and kernite. Cu radiation, Ni filter. Preferred orientation because of cleavage alters the relative intensities of the ezcurrite peaks compared to the data in Table 4.

#### SYNTHESIS

Suhr in his patent (1937) proposed nine ways of preparing the new borate from solution at temperatures above 35°C. Two of these methods were tested during the present study with good results. (1) A solution containing stoichiometric proportions of Na<sub>2</sub>O and B<sub>2</sub>O<sub>3</sub> (10 gr. borax, 2.16 gr. boric acid, 10 gr. water) is boiled until crystallization is nearly complete. It is then diluted with cold water (10 ml) and the crystals separated by filtration. (2) The second method involved saturating the above solution with NaCl, (which greatly enlarges the stability field of the compound), and concentrating above 51°C. The crop of crystals thus obtained was better than by the first method.

Halite was a common mineral at the time of the original deposition of the boron minerals and small veinlets of it may be seen cutting the borax of the Tincalayu deposit. Its presence undoubtedly helped in the crystallization of the ezcurrite. Muessig and Allen (1957) also suggested such a possibility.

For additional information on the synthesis of this and other borates one is referred to Kemp (1956) and Cipriani and Vannunccini (1961).

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