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The Crystal Structure of Ezcurrite

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Abstract

The crystal structure of ezcurrite, a hydrous borate with chemical formula $2Na_2O \cdot 5B_2O_3 \cdot 7H_2O$, has been solved by using direct methods. Three dimensional Weissenberg data (1974 reflections) gave a final *R* factor of 0.050. The lattice parameters are: $a = 8.598$, $b = 9.570$, $c = 6.576$ Å, each ± 0.002; $\alpha = 102°45'$, $\beta = 107°30'$, $\gamma = 71°31'$, each ± 3′. Space group $P\bar{1}$. The crystal structure of ezcurrite contains $[B_5O_7(OH)_3]^{2-}$ polyanions linked together to form chains along *c*. The polyanion is formed by two six-membered boron-oxygen rings, one consisting of two tetrahedra and one triangle, the other of two triangles and one tetrahedron. Therefore, the structural formula of ezcurrite is $[Na_2B_5O_7(OH)_3] \cdot 2H_2O$. The chains are linked together by the Na-polyhedra and by a network of hydrogen bonds.

Introduction

This investigation of the crystal structure of ezcurrite, $2Na_2O \cdot 5B_2O_3 \cdot 7H_2O$, is part of a systematic study of hydrated borate minerals (Dal Negro *et al.*, 1969, 1971). Ezcurrite was described as a new mineral by Muessig and Allen (1957) and restudied by Hurlbut and Aristarain in 1967. Christ (1960) proposed for ezcurrite a $[B_5O_6(OH)_5]^{2-}$ polyanion formed by two tetrahedra and three triangles. The occurrence and the chemical and physical properties of this mineral were fully described by Hurlbut and Aristarain; neither chemical analysis nor redetermination of the unit cell parameters were made on the sample used for this work.

Experimental

A colorless 0.11 × 0.26 × 0.64 mm fragment of ezcurrite from Tincalayu Mine, Salta, Argentina, elongated along [001] was studied. Relevant crystallographic data taken from Hurlbut and Aristarain (1967) are: $P\bar{1}$; $a = 8.598$, $b = 9.570$, $c = 6.576$ Å (each ± 0.002); $\alpha = 102°45'$, $\beta = 107°30'$, $\gamma = 71°31'$ (each ± 3′); $V = 484.7$ Å^3; cell content $Na_4[B_5O_7(OH)_3]_2 \cdot 4H_2O$; specific gravity 2.049 (calc), 2.053 (meas).

Integrated Weissenberg photographs were taken using CuKα radiation and multiple film packs as the crystal was rotated about its *c* axis. Lorentz-polarization and α_1–α_2 spot doubling corrections were applied to the photometrically measured intensities but no correction for absorption ($\mu = 25.5$ cm^{-1}) was made. Within the CuKα limiting sphere, 1974 independent reflections or about 90 percent of the 2198 present were collected. Of these, 893 were too weak to be accurately measured and were considered as unobserved.

Structure Analysis

The structure was solved by the use of direct methods. Sign determination was undertaken by applying the Sayre relationships to 374 reflections with normalized structure factors $|E| > 1.30$. The whole process was performed using a computer program written by R. E. Long (1965). On the F_o-map computed with the phases derived from the set with the highest consistency index, it was possible to find a number of maxima corresponding to the sodium and oxygen atoms. Successive three-dimensional Fourier syntheses, computed on the basis of these coordinates, gave the coordinates of all non-hydrogen atoms in the asymmetric unit. An *R* factor of 0.19 was calculated from these coordinates. This reduced to 0.095 after three least-squares cycles carried out with the Busing, Martin and Levy (1962) computer program ORFLS, using the *f*-curves for neutral atoms of Na, O and B given by Hanson *et al.* (1964) and isotropic thermal parameters. At this stage isotropic convergence was attained and anisotropic thermal parameters were introduced. After two cycles the *R* factor was reduced to 0.06.

A three-dimensional difference Fourier synthesis was computed, giving certain maxima which could be reasonably assigned to hydrogen atoms; however, the number of these peaks was greater than the number of hydrogens present in the unit cell, indicating that some peaks were spurious. Unfortunately it was possible to locate only five hydrogens out of seven because the difference map showed a smearing of the electron density around the two water molecules.

Another cycle of least-squares was undertaken using anisotropic thermal parameters for all non-hydrogen atoms; hydrogens were included in the

TABLE 1. Ezcurrite. Atomic parameters with their standard deviations in parentheses and equivalent isotropic temperature factors after Hamilton (1959).

Atom	x/a	y/b	z/c	B_H(Å^2)
Na(1)	0.8988(2)	0.3700(2)	1.0373(3)	1.81
Na(2)	0.5860(3)	0.1519(3)	0.5616(4)	2.88
O(1)	0.1756(4)	0.2244(3)	0.2169(5)	1.17
O(2)	0.4110(4)	0.0118(3)	0.2130(5)	1.35
O(3)	0.2982(4)	0.1510(3)	-0.0867(5)	1.02
O(4)	0.4508(4)	0.2564(3)	0.2527(5)	1.34
O(5)	0.5666(4)	0.4476(4)	0.2676(5)	1.94
O(6)	0.3627(4)	0.3865(3)	-0.0474(5)	1.34
O(7)	0.0778(4)	0.3727(3)	-0.1868(5)	1.21
O(8)	0.2846(4)	0.2489(3)	-0.3991(5)	1.18
O(9)	-0.2072(4)	0.4156(4)	-0.3508(5)	2.00
O(10)	-0.0113(4)	0.3259(4)	-0.5695(5)	1.47
O(11)	0.8265(5)	0.0547(5)	0.4041(8)	4.07
O(12)	0.8261(7)	0.1440(6)	0.9732(12)	7.28
B(1)	0.3333(7)	0.1608(6)	0.1463(8)	1.05
B(2)	0.2566(7)	0.2850(6)	-0.1777(8)	1.12
B(3)	0.4580(7)	0.3607(6)	0.1560(8)	1.28
B(4)	-0.0485(7)	0.3710(6)	-0.3761(8)	1.14
B(5)	0.1590(6)	0.2623(5)	-0.5812(8)	0.95
H(1)	0.518	-0.045	0.167	3.00
H(2)	0.593	0.507	0.186	3.00
H(3)	-0.292	0.428	-0.481	3.00
H(4)	0.781	-0.021	0.288	5.00
H(5)	0.749	0.087	0.919	5.00

structure factor calculation with isotropic temperature factors equal to 3.0 for those belonging to the hydroxyls and 5.0 for the hydrogens belonging to the water molecules. This reduced the *R* factor to 0.05 for the observed reflections. All the observed structure-factors were weighted equally during the refinement.

The final position parameters are listed in Table 1 and the observed and calculated structure factors are compared in Table 2.[1]

Description and Discussion of the Structure

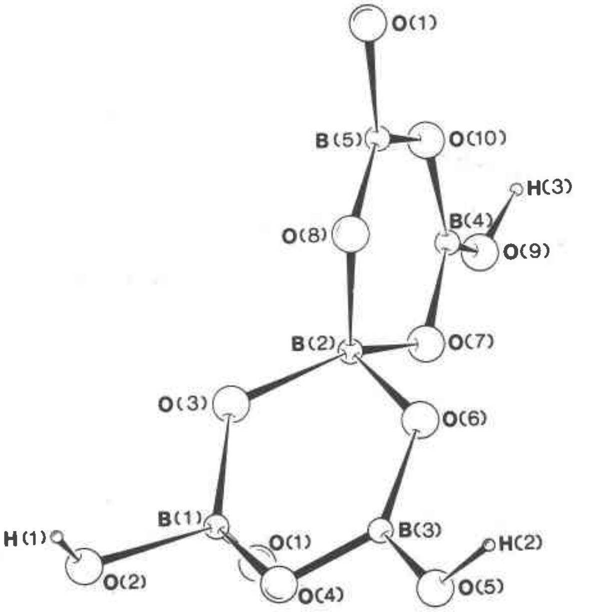

FIG. 1. Projection of the unit $B_5O_7(OH)_3$ on the (100) plane.

The main feature of the crystal structure of ezcurrite is the $[B_5O_7(OH)_3]^{2-}$ unit first found in this structure (Fig. 1). These structural units are connected to form chains along *c;* two equivalent chains are present in a single unit cell (Fig. 2). The $[B_5O_7(OH)_3]^{2-}$ group is formed by two six-membered boron–oxygen rings joined through a common tetrahedral boron. One of the rings consists of two triangles BO_3 and $BO_2(OH)$ in addition to the cen-

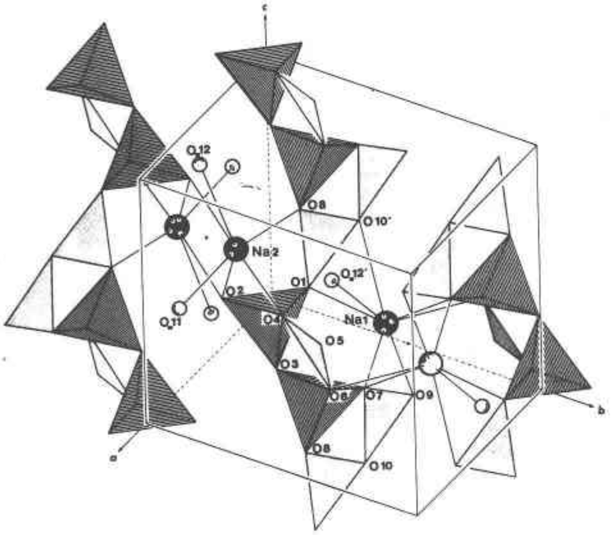

FIG. 2. Clinographic projection of the crystal structure of ezcurrite; the polyanions link together to form chains along *c*.

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[1] To obtain a copy of Table 2, order NAPS Document 01978 from Microfiche Publications, Division of Microfiche Systems Corporation, 305 East 46th Street, New York, N. Y. 10017. Please remit in advance $1.50 for microfiche or $5.00 for photocopies. Check the most recent issue of this journal for the current address and prices.

TABLE 3. Ring angles, planes and deviations from planarity in ezcurrite.

Equation[a] of plane through three oxygens in the form A$\underline{x}$ + B$\underline{y}$ + C$\underline{z}$ = D

Ring 1	A	B	C	D
O(3)-O(4)-O(6)	6.7515	-2.0527	1.2318	2.8285
Ring 2				
O(7)-O(8)-O(10)	3.6254	9.4904	-2.0046	2.1891

Angle between planes 1,2	82°,26

Ring	Atom	Deviation from plane (Å)
1	B(1)	0.504 (5)
	B(2)	-0.668 (5)
	B(3)	0.947 (5)
2	B(2)	-0.202 (6)
	B(4)	-0.095 (6)
	B(5)	0.037 (6)

Angles O-$\hat{B}$-O and B-$\hat{O}$-B internal to the rings.

Ring	Atoms	Angles
1	B(2)-O(3)-B(1)	118°.5
	O(3)-B(1)-O(4)	109°.5
	B(1)-O(4)-B(3)	121°.2
	O(4)-B(3)-O(6)	123°.1
	B(3)-O(6)-B(2)	119°.7
	O(6)-B(2)-O(3)	112°.2
	mean value	117°.4
2	B(2)-O(7)-B(4)	121°.7
	O(7)-B(4)-O(10)	121°.3
	B(4)-O(10)-B(5)	120°.1
	O(10)-B(5)-O(8)	120°.0
	B(5)-O(8)-B(2)	124°.1
	O(8)-B(2)-O(7)	109°.5
	mean value	119°.5

[a] $\underline{x}$,$\underline{y}$,$\underline{z}$ are the atomic coordinates(Å) referred to the crystallographic axes and D is the distance of the planes from the origin(Å).

TABLE 4. Boron-Oxygen, Boron-Boron distances and Oxygen-Boron-Oxygen angles with their standard deviations in parentheses.

B(1)-O(1)	1.468 (6) Å	B(2)-O(3)	1.431 (6) Å
B(1)-O(2)	1.472 (6)	B(2)-O(6)	1.495 (6)
B(1)-O(3)	1.458 (6)	B(2)-O(7)	1.490 (6)
B(1)-O(4)	1.491 (6)	B(2)-O(8)	1.494 (6)
Average	1.472	Average	1.477
B(3)-O(4)	1.322 (6) Å	B(4)-O(7)	1.385 (6) Å
B(3)-O(5)	1.383 (6)	B(4)-O(9)	1.345 (6)
B(3)-O(6)	1.371 (6)	B(4)-O(10)	1.349 (6)
Average	1.359	Average	1.360
B(5)-O(1)	1.336 (6) Å		
B(5)-O(8)	1.349 (6)		
B(5)-O(10)	1.415 (6)		
Average	1.367		

Ring 1		Ring 2	
B(1)-B(2)	2.483 (7) Å	B(2)-B(4)	2.511 (7) Å
B(1)-B(3)	2.454 (7)	B(2)-B(5)	2.512 (7)
B(2)-B(3)	2.480 (7)	B(4)-B(5)	2.395 (8)
Average	2.472	Average	2.472
Between rings 1,2	B(1)-B(5)	2.515 (8) Å	

O(1)-B(1)-O(2)	107°36' (22')	O(3)-B(2)-O(6)	112°12' (23')
O(1)-B(1)-O(3)	110°20' (22')	O(3)-B(2)-O(7)	111°39' (23')
O(1)-B(1)-O(4)	109°30' (22')	O(3)-B(2)-O(8)	110° (23')
O(2)-B(1)-O(3)	110°24' (22')	O(6)-B(2)-O(7)	105°40' (23')
O(2)-B(1)-O(4)	109°22' (22')	O(6)-B(2)-O(8)	107°36' (23')
O(3)-B(1)-O(4)	109°32' (22')	O(7)-B(2)-O(8)	109°31' (23')
O(4)-B(3)-O(5)	117°48' (25')	O(7)-B(4)-O(9)	115° 5' (24')
O(4)-B(3)-O(6)	123° 3' (26')	O(7)-B(4)-O(10)	121°17' (25')
O(5)-B(3)-O(6)	119° 8' (25')	O(9)-B(4)-O(10)	123°37' (25')
O(1)-B(5)-O(8)	126°54' (25')		
O(1)-B(5)-O(10)	113° 2' (22')		
O(8)-B(5)-O(10)	120° 2' (24')		

tral tetrahedron, while the other is formed by one tetrahedron $BO_3(OH)$ and one triangle $BO_2(OH)$ in addition to the central tetrahedron.

According to the fourth rule of Christ (1960), the borate chain in ezcurrite may be considered as the first polymerization product of the still unknown isolated unit $[B_5O_6(OH)_5]^{2-}$, whereas the well-known $[B_5O_8(OH)]^{2-}$ sheets found in $K_2[B_5O_8(OH)]\cdot 2H_2O$ (Marezio, 1969) in veatchite (Clark and Christ, 1971) and in gowerite (Konnert *et al.*, 1972) represent the second step in the polymerization process.

The ezcurrite polyanion and the $[B_5O_8(OH)]^{2-}$ unit are similar, both being built of the same two six-membered rings. In ezcurrite, the mean planes of the two boron–oxygen rings are almost perpendicular, with an angle between the planes of 82.3°. Within the two six-membered rings, the internal B–B distances are nearly equal (2.472 Å is the mean value for the ring 1 and 2.473 Å for the ring 2). The first ring formed by O(7), O(8), O(10) is practically planar with a mean value for the internal angle of 119.5°, the greatest distance from the plane

TABLE 5. Oxygen-Oxygen distances within the B-O polyhedra in Ezcurrite.

Tetrahedron around B(1)		Tetrahedron around B(2)	
O(1)-O(2)	2.373 (4) Å	O(3)-O(6)	2.429 (4) Å
O(1)-O(3)	2.402 (4)	O(3)-O(7)	2.417
O(1)-O(4)	2.417 (4)	O(3)-O(8)	2.396 (4)
O(2)-O(3)	2.406 (4)	O(6)-O(7)	2.379 (4)
O(2)-O(4)	2.418 (4)	O(6)-O(8)	2.412 (4)
O(3)-O(4)	2.409 (4)	O(7)-O(8)	2.437 (4)
Average	2.404	Average	2.411

Triangle around B(3)		Triangle around B(4)	
O(4)-O(5)	2.317 (4) Å	O(7)-O(9)	2.303 (4) Å
O(4)-O(6)	2.368 (4)	O(7)-O(10)	2.383 (4)
O(5)-O(6)	2.374 (4)	O(9)-O(10)	2.374 (4)
Average	2.353	Average	2.353

Triangle around B(5)	
O(1)-O(8)	2.401 (4) Å
O(1)-O(10)	2.295 (4)
O(8)-O(10)	2.394 (4)
Average	2.363

being −0.20 Å (Table 3). In comparison, the second ring built up by O(3), O(4), O(6) is less planar; the mean value for the internal angle is 117.4° and the greatest deviation from the plane is +0.95 Å. The polyanions link together to form chains with an angle B(5)–O(1)–B(1) of 127.5° and a separation B(5)–B(1) of 2.515 Å. The B–O distances in the ezcurrite polyanion (Table 4) are in good agreement with those found in the literature for other borates. The mean B–O bond length is 1.475 Å for the tetrahedral boron and 1.362 Å for triangular coordination. However, appreciable deviations from the average indicate that the B–O polyhedra are somewhat distorted. The O–B–O angles fall within the expected range (Table 4), as do the O–O distances (Table 5).

There are two independent sodium atoms in the ezcurrite structure (Fig.3), one being six-coordinated and the other seven. The sodium cations bond to the oxygen anions in two different centrosymmetrically related borate chains. Moreover, each Na–polyhedron shares an edge with another Na–polyhedron and the centrosymmetric pairs so formed link together through the vertex O(12) to form Na–O chains running along [111].

TABLE 6. Sodium coordinates in Ezcurrite.

Oxygen atom	Coordinates of oxygen atom[a]			Na(1)-O distance(Å)
O(1)	1.1755	0.2244	1.2169	2.436 (3)
O(6)	0.6373	0.6135	1.0474	2.681 (4)
O(7)	1.0778	0.3727	0.8132	2.438 (4)
O(7')	0.9222	0.6273	1.1868	2.489 (4)
O(9)	0.7928	0.4156	0.6492	2.525 (4)
O(10)	0.9887	0.3259	1.4305	2.557 (4)
O(12)	0.8261	0.1440	0.9732	2.355 (6)
				Na(2)-O distance(Å)
O(2)	0.4110	0.0118	0.2130	2.676 (4)
O(2')	0.5890	-0.0118	0.7870	2.373 (4)
O(4)	0.4508	0.2564	0.2527	2.284 (4)
O(8)	0.2845	0.2489	0.6009	2.533 (4)
O(11)	0.8265	0.0547	0.4041	2.420 (5)
O(12)	0.8261	0.1440	0.9732	2.868 (6)

[a] Sodium atoms at x,y,z in Table 1.

The two water molecules in the asymmetric unit are bonded only to the sodium atoms. In particular the water molecule O(11) is linked to Na(2) while O(12) connects the two independent Na–O polyhedra. The Na–O bond lengths range from 2.281 to 2.858 Å (Table 6). It has been possible to single out the hydrogens belonging to three hydroxyls (but

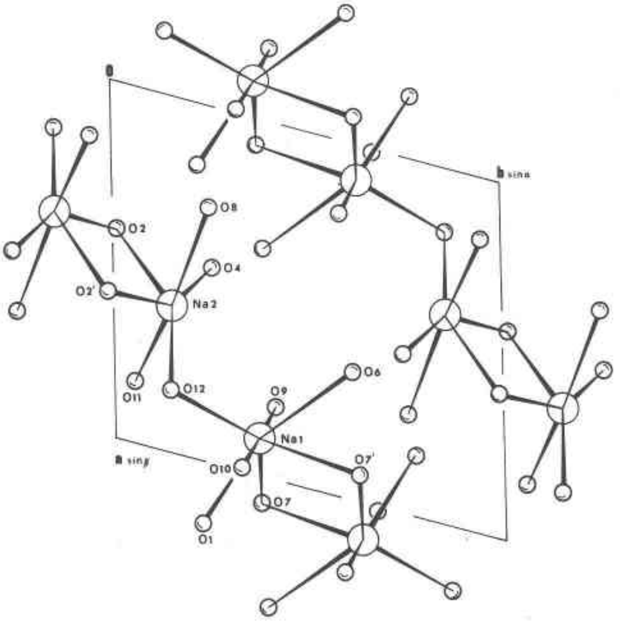

FIG. 3. Details of the projection on the (001) plane showing the Na-O chains.

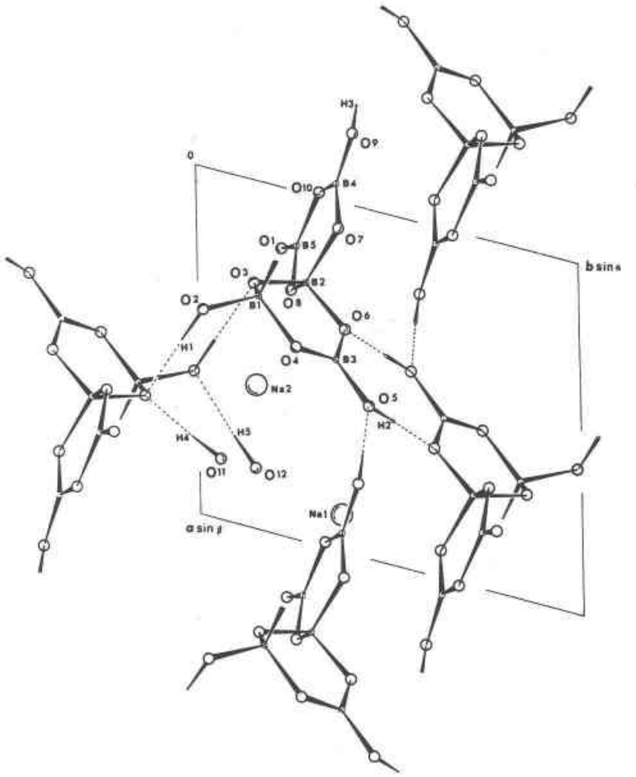

FIG. 4. Projection on the (001) plane showing the hydrogen bonds.

TABLE 7. Distances related to the hydrogen bonds in Ezcurrite.

Donor[a]	Hydrogen	Receptor	Coordinates of receptor			O-H distance	O-O distance
O(2)	H(1)	O(3)	0.702	−0.151	0.087	1.01 Å	2.752 (4) Å
O(5)	H(2)	O(6)	0.637	0.613	0.047	0.98	2.672 (4)
O(9)	H(3)	O(5)	−0.434	0.448	−0.732	0.95	2.683 (4)
O_w(11)	H(4)	O(3)	0.702	−0.151	0.087	1.01	2.750 (5)
O_w(12)	H(5)	O(2)	0.589	−0.012	0.787	0.93	2.739 (6)

[a] Donor atoms taken at x,y,z in Table 1.

only one hydrogen for each water molecule). On the other hand there are only two O–O distances less than 3.1 Å involving the two water molecules, suggesting that two hydrogens do not form hydrogen bonds. Each B–O chain links to four other chains through the hydrogens belonging to the hydroxyl groups. The chain (Fig. 4) formed by the polyanion with atomic coordinates *x, y, z* of Table 1 is connected to two centrosymmetric chains through pairs of centrosymmetric hydrogen bonds. Two other chains, related by translations along *a* and *c* (atomic coordinates $1 + x, y, 1 + z$ and $x - 1, y, z - 1$), are linked to the first chain through one distinct hydrogen bond. In this way there is a thick network of hydrogen bonds which provides the connections among the polyanions and, through the water molecules, among polyanions and Na–polyhedra. The O–O distances involved in hydrogen-bonding and the O–H bond lengths are listed in Table 7.

TABLE 8. Analysis of the anisotropic thermal parameters in ezcurrite[a]

Atom	r.m.s.	$U_i\underline{a}$	$U_i\underline{b}$	$U_i\underline{c}$	Atom	r.m.s.	$U_i\underline{a}$	$U_i\underline{b}$	$U_i\underline{c}$
Na(1)	0.127(4)	121	81	129	O(4)	0.092(7)	120	96	131
	0.162(12)	42	32	120		0.120(7)	60	18	119
	0.162(11)	58	114	129		0.167(5)	134	72	55
Na(2)	0.115(4)	102	65	142	O(5)	0.102(8)	143	118	103
	0.183(4)	13	70	120		0.122(7)	60	18	119
	0.250(3)	95	147	109		0.220(5)	123	65	52
B(1)	0.090(13)	86	136	48	O(6)	0.093(8)	142	123	101
	0.120(11)	119	70	41		0.110(7)	88	37	130
	0.133(11)	150	126	91		0.174(5)	127	75	42
B(2)	0.088(13)	135	63	77	O(7)	0.062(11)	121	68	120
	0.122(11)	54	49	67		0.125(6)	115	72	36
	0.141(11)	65	51	154		0.162(6)	137	151	72
B(3)	0.114(11)	159	96	91	O(8)	0.078(9)	104	74	144
	0.125(11)	74	13	115		0.127(6)	29	91	125
	0.142(10)	76	102	154		0.151(6)	115	164	88
B(4)	0.114(9)	135	82	113	O(9)	0.087(8)	130	86	120
	0.117(11)	46	45	139		0.133(7)	47	89	146
	0.128(10)	84	134	120		0.225(5)	110	176	79
B(5)	0.070(17)	105	173	83	O(10)	0.103(7)	134	81	114
	0.103(11)	52	92	56		0.123(7)	123	86	27
	0.144(10)	137	95	34		0.173(6)	117	170	77
O(1)	0.088(8)	115	69	129	O(11)	0.134(8)	141	115	107
	0.116(7)	29	73	137		0.202(6)	110	39	82
	0.152(6)	104	153	103		0.310(6)	58	61	161
O(2)	0.101(7)	61	125	69	O(12)	0.131(10)	138	138	93
	0.127(7)	28	44	117		0.266(7)	111	48	113
	0.158(6)	85	112	144		0.435(8)	57	89	156
O(3)	0.085(8)	125	85	125					
	0.116(7)	77	11	91					
	0.135(6)	141	100	35					

[a] Root mean square thermal vibrations along the ellipsoid axes(Å) and angles (°) between the crystallographic axes and the principal axes (U_i) of the vibration ellipsoid.

Analysis of the anisotropic thermal parameters (Table 8) reveals that the thermal anisotropy of the atoms in ezcurrite is not large except for Na(2) and the water molecules. Taking into account the great anisotropy of Na(2) with respect to Na(1), the strong thermal motion of the O(12) water molecule can probably be explained as interaction between O(12) and Na(2), the major axis of the ellipsoid of thermal vibration for both atoms being localized along the bond direction.

Considering electrostatic balance, values ranging from 1.90 to 2.10 valence units are found for each oxygen atom on the basis of the correlations between bond length and bond strength given by Zachariasen (1963) for B–O and O–H . . . O bonds. Strengths of $1/7 = 0.142$ and $1/6 = 0.166$ valence units have been assigned to each oxygen atom coordinated to Na(1) and Na(2), respectively. One proton for each water molecule does not appear to form any hydrogen bonds, and for this reason a 1.0 valence was assigned to it.

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