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Key indicators

Single-crystal X-ray study
T = 173 K
Mean $\sigma(\text{C}-\text{C}) = 0.007 \text{ \AA}$
Disorder in main residue
R factor = 0.060
wR factor = 0.163
Data-to-parameter ratio = 15.9

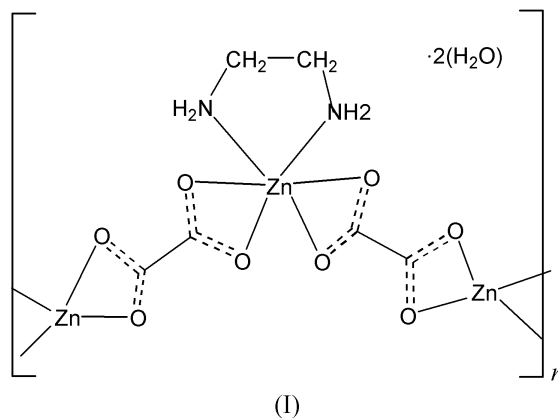
For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.

catena-Poly[[[(ethylenediamine- κ^2N,N')zinc(II)]- μ -oxalato] dihydrate]

In the crystal structure of the title compound, $\{[\text{Zn}(\text{C}_2\text{O}_4)(\text{C}_2\text{H}_8\text{N}_2)] \cdot 2\text{H}_2\text{O}\}_n$, the Zn^{II} atom adopts an octahedral geometry, formed by two N atoms of an ethylenediamine ligand [$\text{Zn}-\text{N} = 2.112(4)-2.124(4) \text{ \AA}$] and four O atoms of two bridging oxalate ligands [$\text{Zn}-\text{O} = 2.105(3)-2.154(3) \text{ \AA}$]. The asymmetric unit consists of one and one-half formula units.

Comment

The title compound, (I), was obtained by *in situ* synthesis during the preparation of a zinc(II) compound with the ligand 2,2'-biimidazole (abbreviated as biz) and ammonium thiocyanate. The ligand biz is known to hydrolyse *in situ* to form new organic ligands (Woodburn & O'Gee 1952; Wang & Bauman, 1965; van Albada *et al.*, 2003). In this study, it has been observed that the biz ligand hydrolyses to give ethylenediamine molecules and oxalate anions, a type of hydrolysis which was already reported in the literature (Woodburn & O'Gee, 1952).



The Zn atom adopts an octahedral geometry, formed by two N atoms of an ethylenediamine ligand [$\text{Zn}-\text{N} = 2.112(4)-2.124(4) \text{ \AA}$] and four O atoms of two bridging oxalate ligands [$\text{Zn}-\text{O} = 2.105(3)-2.154(3) \text{ \AA}$, see Table 1 for details], forming a polymeric one-dimensional array of Zn-oxalate units (Fig. 1). Such a polymeric Zn^{II} -oxalate array has only been reported once previously, with the ligand 2-methylimidazole (Jansen *et al.*, 1979). The asymmetric unit consists of one and one-half formula units, one Zn atom lying on a twofold rotation axis.

The crystal structure of (I) is stabilized by intricate intermolecular hydrogen bonding between the amine N atoms and the O atoms of the oxalate ligand and the water molecules, with $\text{N} \cdots \text{O}$ distances in the range $3.121(6)-3.210(6) \text{ \AA}$, and

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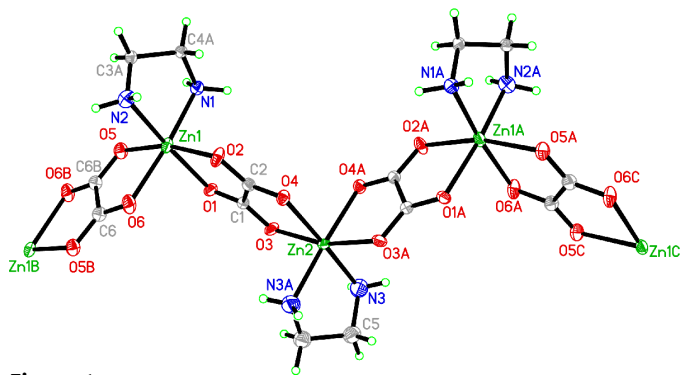


Figure 1

Part of the molecular structure of (I), showing 50% probability displacement ellipsoids. The non-coordinating water molecules have been omitted for clarity. Only one site of the disordered ethylenediamine molecule is shown. Atom labels with suffixes *A*, *B* and *C* are generated by the symmetry codes $(1-x, y, \frac{3}{2}-z)$, $(\frac{1}{2}-x, \frac{1}{2}-y, 1-z)$ and $(\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z)$, respectively.

between the O atoms of the oxalate ligand and the O atoms of the water molecules, with O...O distances ranging from 2.736 (6)–2.895 (4) Å (Table 2).

Experimental

The ligand 2,2'-biimidazole (*biz*) was prepared according to a method reported in the literature (Wang & Bauman, 1965). The title Zn complex was obtained by Zn-induced hydrolysis of the *biz* ligand. The title compound was synthesized by mixing *biz* (2 mmol) and zinc(II) perchlorate hexahydrate (1 mmol) in an ethanol solution. To the resulting solution, an aqueous solution (3 ml) of ammonium thiocyanate (2 mmol) was added dropwise, and the mixture was boiled for about 1 h. It was then filtered and left to stand at room temperature. After about one month, white crystals were obtained, which were recrystallized from an ethanol–acetonitrile (1:1) solution (yield 66%). Elemental analysis [found (calculated)] for $C_{12}H_{36}N_6O_{18}Zn_3$: C 19.5 (19.3), H 4.9 (4.8), N 11.4% (11.2%). In the IR spectrum, the OH and NH vibrations were observed at 3488, 3410 and 3361 cm^{-1} , while the C=O vibration of the oxalate ligand was observed at 1602 cm^{-1} .

Crystal data

$[Zn(C_2O_4)(C_2H_8N_2)] \cdot 2H_2O$
 $M_r = 249.53$
 Monoclinic, $C2/c$
 $a = 25.757$ (5) Å
 $b = 9.053$ (3) Å
 $c = 11.623$ (2) Å
 $\beta = 93.05$ (3)°
 $V = 2706.4$ (11) Å³
 $Z = 12$

$D_x = 1.837$ Mg m⁻³
 Mo $K\alpha$ radiation
 Cell parameters from 20 214 reflections
 $\theta = 2.9$ – 27.6 °
 $\mu = 2.73$ mm⁻¹
 $T = 173$ (2) K
 Prism, colorless
 $0.20 \times 0.12 \times 0.10$ mm

Data collection

Nonius KappaCCD area-detector diffractometer
 φ and ω scans
 Absorption correction: multi-scan (SADABS; Sheldrick, 1996)
 $T_{min} = 0.606$, $T_{max} = 0.764$
 20 214 measured reflections

3121 independent reflections
 2425 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.044$
 $\theta_{max} = 27.6$ °
 $h = -33 \rightarrow 33$
 $k = -11 \rightarrow 11$
 $l = -14 \rightarrow 15$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.060$
 $wR(F^2) = 0.163$
 $S = 1.23$
 3121 reflections
 196 parameters
 H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.0528P)^2 + 25.3123P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{max} = 0.001$
 $\Delta\rho_{max} = 1.47$ e Å⁻³
 $\Delta\rho_{min} = -2.17$ e Å⁻³

Table 1

Selected geometric parameters (Å, °).

Zn1–O2	2.105 (3)	Zn1–O1	2.154 (3)
Zn1–N2	2.112 (4)	Zn2–N3	2.124 (4)
Zn1–N1	2.116 (4)	Zn2–O3	2.127 (3)
Zn1–O5	2.116 (3)	Zn2–O4	2.148 (3)
Zn1–O6	2.149 (3)		
O2–Zn1–O5	165.94 (14)	O3–Zn2–O3 ⁱ	164.82 (17)
N1–Zn1–O6	173.48 (14)	N3–Zn2–O4	176.95 (15)
N2–Zn1–O1	173.90 (15)		

Symmetry code: (i) $1-x, y, \frac{3}{2}-z$.

Table 2

Hydrogen-bonding geometry (Å, °).

<i>D</i> –H... <i>A</i>	<i>D</i> –H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> –H... <i>A</i>
N1–H11A...O8	0.92	2.29	3.174 (5)	160
N1–H11B...O8	0.92	2.46	3.174 (5)	135
N1–H12A...O9 ^{vi}	0.92	2.29	3.133 (6)	153
N1–H12B...O9 ^{vi}	0.92	2.42	3.133 (6)	135
N2–H21A...O9 ⁱⁱ	0.92	2.27	3.166 (7)	165
N2–H21B...O9 ⁱⁱ	0.92	2.46	3.166 (7)	134
N2–H22A...O8 ⁱⁱⁱ	0.92	2.36	3.210 (6)	154
N2–H22B...O8 ⁱⁱⁱ	0.92	2.46	3.210 (6)	139
N3–H31...O10 ^v	0.83	2.36	3.121 (6)	153
N3–H32...O10 ^v	0.93	2.35	3.147 (6)	144
O8–H81...O4 ⁱ	0.84	2.08	2.895 (4)	166
O8–H82...O2 ^{vii}	0.87	1.89	2.747 (5)	168
O9–H91...O5 ^v	0.84	1.92	2.736 (6)	164
O9–H92...O1	0.84	2.05	2.852 (6)	159
O10–H101...O3 ^{vi}	0.84	1.98	2.757 (6)	152
O10–H102...O6 ⁱⁱ	0.84	2.00	2.832 (5)	167

Symmetry codes: (i) $1-x, y, \frac{3}{2}-z$; (ii) $\frac{1}{2}-x, \frac{1}{2}-y, 1-z$; (iii) $x, -y, z-\frac{1}{2}$; (iv) $\frac{1}{2}+x, \frac{1}{2}-y, \frac{1}{2}+z$; (v) $\frac{1}{2}-x, \frac{1}{2}+y, \frac{3}{2}-z$; (vi) $\frac{1}{2}-x, y-\frac{1}{2}, \frac{3}{2}-z$; (vii) $x, -y, \frac{1}{2}+z$.

One of the ethylenediamine ligands was found to be disordered over two conformations. The occupancies of the two conformers were initially refined to 0.56 (1) and 0.44 (1), respectively, but later fixed at 0.50 each. The C- and N-bound H atoms were placed in calculated positions (N–H = 0.92 Å and C–H = 0.99 Å) and allowed to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(\text{parent atom})$. The H atoms of the water molecules were located in a difference map and their positions were not refined but their $U_{iso}(H)$ values were set equal to $1.5U_{eq}(O)$.

Data collection: COLLECT (Nonius, 2002); cell refinement: COLLECT and DENZO/SCALEPACK (Otwinowski & Minor, 1997); data reduction: COLLECT and DENZO/SCALEPACK; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); software used to prepare material for publication: SHELXTL.

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