Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters ($\mathring{A}^2$)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Cu	0	0	1/2	0.03582 (14)
O1	−0.1718 (2)	−0.0327 (2)	0.5160 (2)	0.0454 (5)
O2	−0.0870 (2)	−0.1477 (2)	0.6634 (2)	0.0567 (5)
O3	−0.2191 (2)	−0.3281 (3)	0.7476 (2)	0.0686 (7)
C1	−0.1792 (2)	−0.1181 (3)	0.5926 (2)	0.0410 (6)
C2	−0.3009 (2)	−0.1887 (3)	0.5925 (2)	0.0382 (6)
C3	−0.3136 (3)	−0.2918 (3)	0.6677 (2)	0.0455 (6)
C4	−0.4260 (3)	−0.3623 (4)	0.6614 (3)	0.0629 (9)
C5	−0.5240 (3)	−0.3293 (4)	0.5827 (3)	0.0741 (11)
C6	−0.5141 (3)	−0.2266 (5)	0.5084 (3)	0.0761 (12)
C7	−0.4025 (3)	−0.1582 (4)	0.5129 (3)	0.0556 (8)
O4	−0.2272 (2)	−0.5680 (2)	0.2826 (2)	0.0694 (7)
N1	0.0096 (2)	−0.1893 (2)	0.4352 (2)	0.0376 (5)
N2	−0.3092 (2)	−0.3592 (3)	0.2452 (2)	0.0556 (7)
C8	−0.0945 (2)	−0.2486 (2)	0.3817 (2)	0.0353 (6)
C9	−0.0969 (2)	−0.3783 (2)	0.3381 (2)	0.0375 (6)
C10	0.0139 (3)	−0.4515 (3)	0.3506 (3)	0.0554 (8)
C11	0.1216 (3)	−0.3913 (3)	0.4036 (3)	0.0615 (9)
C12	0.1156 (3)	−0.2616 (3)	0.4449 (3)	0.0488 (7)
C13	−0.2169 (3)	−0.4432 (3)	0.2852 (2)	0.0457 (7)

Table 2. *Selected geometric parameters ($\mathring{A}$, °)*

Cu—O1	1.928 (2)	O3—C3	1.354 (3)
Cu—N1	2.022 (2)	C1—C2	1.487 (3)
Cu—O2	2.766 (2)	O4—C13	1.222 (3)
O1—C1	1.275 (3)	N2—C13	1.327 (4)
O2—C1	1.255 (3)	C9—C13	1.500 (3)
O1—Cu—N1	90.01 (8)	O3—C3—C4	117.8 (3)
O1—Cu—O2	52.72 (7)	O3—C3—C2	122.4 (3)
N1—Cu—O2	81.91 (7)	C8—C9—C13	121.4 (2)
O2—C1—O1	122.3 (2)	C10—C9—C13	120.6 (2)
O2—C1—C2	120.2 (3)	O4—C13—N2	122.9 (3)
O1—C1—C2	117.4 (2)	O4—C13—C9	120.1 (3)
C7—C2—C1	120.3 (2)	N2—C13—C9	117.0 (2)
C3—C2—C1	120.9 (2)		

Table 3. *Hydrogen-bonding geometry ($\mathring{A}$, °)*

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H10$\cdots$O4^i	0.83 (3)	2.07 (3)	2.895 (3)	173 (3)
N2—H11$\cdots$O2^{ii}	0.80 (3)	2.30 (3)	3.022 (3)	150 (3)
O3—H5$\cdots$O2	0.87 (4)	1.79 (4)	2.588 (3)	151 (3)

Symmetry codes: (i) $-\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; (ii) $x - \frac{1}{2}, -\frac{1}{2} - y, z - \frac{1}{2}$.

The space group $P2_1/n$ was deduced from the systematic absences. All H atoms were found in the difference electron-density map and their positional and isotropic displacement parameters were fully refined. Final difference electron-density map revealed a maximum and minimum at 0.272 and −0.244 e $\mathring{A}^{-3}$, respectively. Calculations were performed on the standard PC 486/16MB.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *ORTEPII* (Johnson, 1971) and *PLUTON* (Spek, 1991). Software used to prepare material for publication: *SHELXL93*.

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Diaquabis(dimethyl 3-oxopentanedioato-O^1,O^3)zinc(II)

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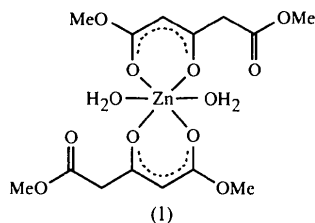
Abstract

The molecular structure of the title compound, $[Zn(C_7H_9O_5)_2(H_2O)_2]$, shows the Zn atom coordinated by two monoanionic bidentate enolate ligands and two O atoms of water resulting in a pseudo-octahedral arrangement. The H atoms of the water molecules are oriented in such a way that one H atom forms an intramolecular hydrogen bond [H6$B\cdots$O2($-x, -y, -z$)], while the other H atom forms an intermolecular hydrogen bond [H6$A\cdots$O3($1 - x, -y, -z$)]. The intermolecular contacts link the molecules into stacks.

Comment

We have shown that esters of 3-oxopentanedioic acid RCH_2COCH_2R, (2), react with carbonato complexes

[Pt(CO₃)L₂] in warm ethanol to afford the oxatrimethyl-enemethane complexes [Pt{*n-R*CHC(O)CH*R*}L₂] (*R* = CO₂Me, CO₂Et, CO₂^{n}Pr; *L* = tertiary phosphine or arsine donor ligand) (Clarke *et al.*, 1984) which contain the formally dianionic ligand [*R*CHCOCH*R*]²⁻. We now find that the dimethyl ester of (2) reacts with aqueous zinc chloride to afford the title monoanionic enolate complex, (1).

(1)

The Zn atom is located on an inversion centre with approximately octahedral coordination completed by two bidentate dimethyl 3-oxopentanedioato ligands and two water molecules (Fig. 1). The O3—Zn—O4 angle of the bidentate ligand is 89.23 (8)° and the angles between the coordinating O atoms of the ligand and that of the water molecule are 93.95 (8)° for O3—Zn—O6 and 88.70 (8)° for O4—Zn—O6. The Zn—O bond distances are shorter for the ligand atoms O3 and O4 [2.019 (2) and 2.079 (2) Å, respectively] than for O6 of the water molecule [2.178 (2) Å]. The Zn—O distances are comparable with those found in aquabis(methylmaltolato)zinc and aquabis(3-hydroxy-1,2-dimethylpyridinone)zinc of 2.006–2.264 Å (Burgess, Fawcett, Parsons, Ahmed & Russell, 1997). The orientation of the water molecule is such that one of the H atoms forms an intramolecular hydrogen bond to the O2 atom of the ligand [H6*B*···O2^i 2.141 (3) and O6···O2^i

3.045 (4) Å; symmetry code: (i) −*x*, −*y*, −*z*], while the other H atom forms an intermolecular hydrogen bond to an O3 atom of an adjacent molecule [H6*A*···O3^{ii} 2.128 (3) and O6···O3^{ii} 2.754 (3) Å; symmetry code: (ii) 1 − *x*, −*y*, −*z*]. The intermolecular hydrogen bonds link the molecules in stacks down the *a* axis with two hydrogen bonds H6*A*···O3^{ii} and O3···H6*A*^{ii} between each adjacent molecule.

Experimental

Basic zinc carbonate (0.68 g, 2 × 10⁻³ mol) was suspended in water (9 ml) and was converted to the chloride by the gradual addition of 6 *M* HCl until the solution was just acidic. Dimethyl 3-oxopentanedioate (3.48 g, 0.02 mol) was suspended in water (10 ml) and 6 *M* ammonia added dropwise with stirring. The resulting solution was added with stirring to the zinc chloride solution. The white precipitate was filtered and washed with water and dried in air to give 0.78 g of the title enolato complex. Colourless crystals were obtained from chloroform.

Crystal data

[Zn(C₇H₉O₅)₂(H₂O)₂]	Mo *K*α radiation
*M*ᵣ = 447.69	λ = 0.71073 Å
Monoclinic	Cell parameters from 23
*P*2₁/*c*	reflections
a = 4.939 (2) Å	θ = 6.4–12.5°
b = 20.110 (6) Å	μ = 1.309 mm⁻¹
c = 10.003 (3) Å	*T* = 293 (2) K
β = 98.85 (2)°	Block
V = 981.7 (6) Å³	0.45 × 0.24 × 0.19 mm
Z = 2	Colourless
*D*ₓ = 1.515 Mg m⁻³	
*D*ₘ not measured	

Data collection

Siemens *P*4 diffractometer	*R*ᵢₙₜ = 0.034
ω scans	θₘₐₓ = 25°
Absorption correction:	*h* = −1 → 5
semi-empirical on ψ scans	*k* = −1 → 23
(Sheldrick, 1990)	*l* = −11 → 11
*T*ₘᵢₙ = 0.786, *T*ₘₐₓ = 0.837	3 standard reflections
2473 measured reflections	every 100 reflections
1710 independent reflections	intensity variation: <1%
1323 reflections with	
I > 2σ(*I*)	

Refinement

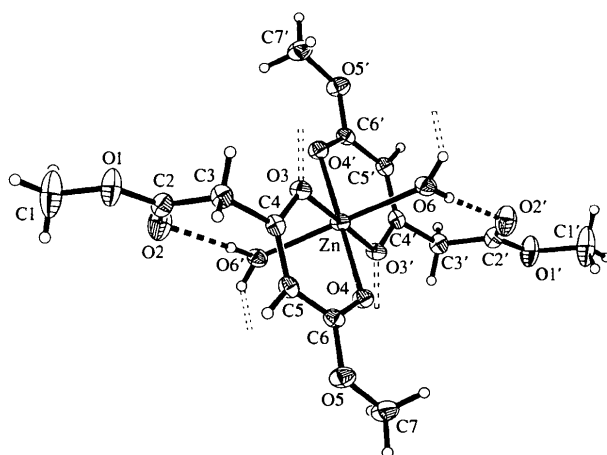|	
Refinement on *F*²	(Δ/σ)ₘₐₓ = −0.001
R[*F*² > 2σ(*F*²)] = 0.034	Δρₘₐₓ = 0.26 e Å⁻³
wR(*F*²) = 0.092	Δρₘᵢₙ = −0.31 e Å⁻³
S = 1.061	Extinction correction: none
1709 reflections	Scattering factors from
124 parameters	*International Tables for*
H atoms: see below	*Crystallography* (Vol. C)
w = 1/[σ²(*F*ₒ²) + (0.0368*P*)²	
+ 0.4667*P*]	
where *P* = (*F*ₒ² + 2*F*c²)/3	

Fig. 1. Molecular structure of the title compound showing the atom-numbering scheme and 30% displacement ellipsoids. Primed atoms are generated by a centre of symmetry. The solid dashed lines indicate intramolecular hydrogen bonding, while intermolecular hydrogen bonds are shown as open dashed lines indicating their direction.

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Zn	0	0	0	0.0305 (2)
O1	0.1113 (7)	−0.20833 (12)	−0.4191 (3)	0.0671 (8)
O2	−0.1124 (6)	−0.17403 (13)	−0.2557 (3)	0.0643 (8)
O3	0.1932 (4)	−0.03891 (11)	−0.1458 (2)	0.0345 (5)
O4	−0.2321 (4)	0.05893 (10)	−0.1455 (2)	0.0353 (5)
O5	−0.4742 (5)	0.07375 (12)	−0.3510 (2)	0.0445 (6)
O6	0.2876 (4)	0.08217 (11)	0.0426 (2)	0.0377 (5)
C1	−0.0154 (13)	−0.2730 (2)	−0.4155 (6)	0.104 (2)
C2	0.0427 (7)	−0.1635 (2)	−0.3339 (3)	0.0405 (8)
C3	0.1857 (7)	−0.0987 (2)	−0.3502 (3)	0.0376 (7)
C4	0.0749 (6)	−0.0445 (2)	−0.2694 (3)	0.0306 (7)
C5	−0.1453 (6)	−0.0087 (2)	−0.3312 (3)	0.0331 (7)
C6	−0.2773 (6)	0.0421 (2)	−0.2661 (3)	0.0316 (7)
C7	−0.6237 (8)	0.1246 (2)	−0.2945 (4)	0.0535 (10)

Table 2. *Selected geometric parameters (Å, °)*

Zn—O3	2.019 (2)	O3—C4	1.289 (3)
Zn—O4	2.079 (2)	O4—C6	1.240 (3)
Zn—O6	2.178 (2)	O5—C6	1.349 (4)
O1—C2	1.320 (4)	O5—C7	1.428 (4)
O1—C1	1.446 (5)	O6· · ·O2^i	3.045 (4)
O2—C2	1.196 (4)	O6· · ·O3^{ii}	2.754 (3)
O3—Zn—O4	89.23 (8)	O4—Zn—O6	88.70 (8)
O3—Zn—O6	93.95 (8)		

Symmetry codes: (i) $-x, -y, -z$; (ii) $1 - x, -y, -z$.

Aqua H atoms were located from a difference Fourier map and thereafter allowed to ride on O6. All other H atoms were introduced at geometrically calculated positions and allowed to ride on their parent atoms.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Software used to prepare material for publication: *SHELXTL/PC*.

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