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Redetermination of *cis*-diaqua-diglycolatozinc(II)

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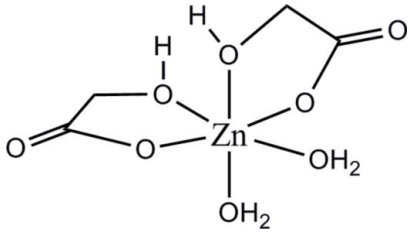
Key indicators: single-crystal X-ray study; T = 273 K; mean σ(C–C) = 0.003 Å; R factor = 0.030; wR factor = 0.072; data-to-parameter ratio = 17.7.

The title complex, $[Zn(C_2H_3O_3)_2(H_2O)_2]$, was prepared and the crystal structure determined as part of a ^{67}Zn solid state nuclear magnetic resonance study. In the title complex, the Zn atom has a disorted octahedral coordination comprising two bidentate glycolate ligands and two water molecules. The water molecules are *cis* to each other; one is *trans* to a carboxylate O atom and the other *trans* to an alcohol O atom. The crystal structure has an extensive O—H···O hydrogen-bond network.

Related literature

The crystal structure of the title complex was first reported by Fischinger & Webb (1969). For bond-length data, see: Allen *et al.* (1987).



Experimental

Crystal data

$[Zn(C_2H_3O_3)_2(H_2O)_2]$ M_r = 251.49
Monoclinic, $P2_1/c$
a = 11.391 (2) Å
b = 5.857 (1) Å
c = 12.511 (2) Å
β = 91.198 (9)°
V = 834.5 (2) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.96 mm^{-1}
T = 273 (2) K
0.3 × 0.3 × 0.1 mm

Data collection

Bruker P4 diffractometer
Absorption correction: ψ scan (*SADABS*; Bruker, 2000)
T_{min} = 0.425, T_{max} = 0.744
2102 measured reflections
2102 independent reflections
1751 reflections with $I > 2\sigma(I)$
3 standard reflections
every 97 reflections
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.072
S = 0.99
2102 reflections
119 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.45 e Å^{-3}
$\Delta\rho_{min}$ = −0.51 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
O1—H8···O4^{i}	0.90	1.93	2.821 (3)	167
O1—H7···O8^{ii}	0.88	1.84	2.716 (3)	174
O2—H6···O5^{i}	0.91	1.82	2.697 (3)	161
O2—H5···O5^{iii}	0.85	1.88	2.688 (3)	158
O6—H4···O8^{iv}	0.84	1.82	2.665 (2)	177
O3—H3···O7^{iv}	0.83	1.97	2.761 (3)	160

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

Data collection: *XSCANS* (Bruker, 2000); cell refinement: *XSCANS*; data reduction: *XSCANS*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *SHELXTL* (Sheldrick, 2008); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SU2079).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2000). *XSCANS* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
Fischinger, A. J. & Webb, L. E. (1969). *Chem. Commun.* pp. 407–408.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.

supporting information

Acta Cryst. (2008). E**64**, m1635 [doi:10.1107/S1600536808039585]

Redetermination of *cis*-diaquadiglycolatozinc(II)

Paul Kennedy, Neferterneken Francis, David Rovnyak and Margaret E. Kastner

S1. Comment

As part of a Zinc-67 solid state nuclear magnetic resonance study the title complex, (I), was prepared and the crystal structure determined. The structure of this complex was first report by Fischinger & Webb (1969) but no fractional crystal coordinates were reported.

The molecular structure of (I) is illustrated in Fig. 1. The zinc atom has a distorted octahedral coordination sphere composed of two bidentate gylcolato ligands and two water molecules. The water molecules are *cis* to each other; one (O1) is *trans* to a carboxylate O-atom (O4), and the other, (O2), is *trans* to an alcohol O-atom (O6). The bond distances and angles are normal for zinc(II) complexes (Allen *et al.*, 1987)

In the crystal structure of (I) there in an extensive O—H···O hydrogen bonding network (Table 1). The two water molecules (O1 and O2) bond to the two oxygens (O4 and O5) of a carboxylate group related by the c-glide. The two alcohol groups (O3 and O6) form hydrogen bonds with the other carboxylate group (atoms O7 and O8) translated by one unit cell along the b axis. The carbonyl oxygen O8 of this ligand also makes a two dimensional hydrogen bonded network with one of the waters (O1) around the inversion center. The other water molecule, (O2), forms an H-bond with a carbonyl oxygen (O5) related by the 2-fold screw axis.

S2. Experimental

Glycolic acid (100 mg, 3 mmol), purchased from Sigma-Aldrich (99%), was dissolved in 5 ml of deionized water. Basic zinc carbonate (80 mg, 2 mmol) was added and the mixture stirred for 10 minutes while heating to ca. 60°C. The resulting mixture was filtered, and the filtrate left to stand at room temperature until large needle-like crystals grew by slow evaporation of the water.

S3. Refinement

The alcohol and water H-atoms were placed at the locations identified in a difference Fourier map and were held fixed, with U_{iso}(H) set to 0.05 A^2: O-H = 0.8293 - 0.9135 Å. The C-bound H-atoms were included in calculated positions and treated as riding atoms: C-H = 0.97 Å with U_{iso}(H) = 1.2U_{eq}(parent C-atom).

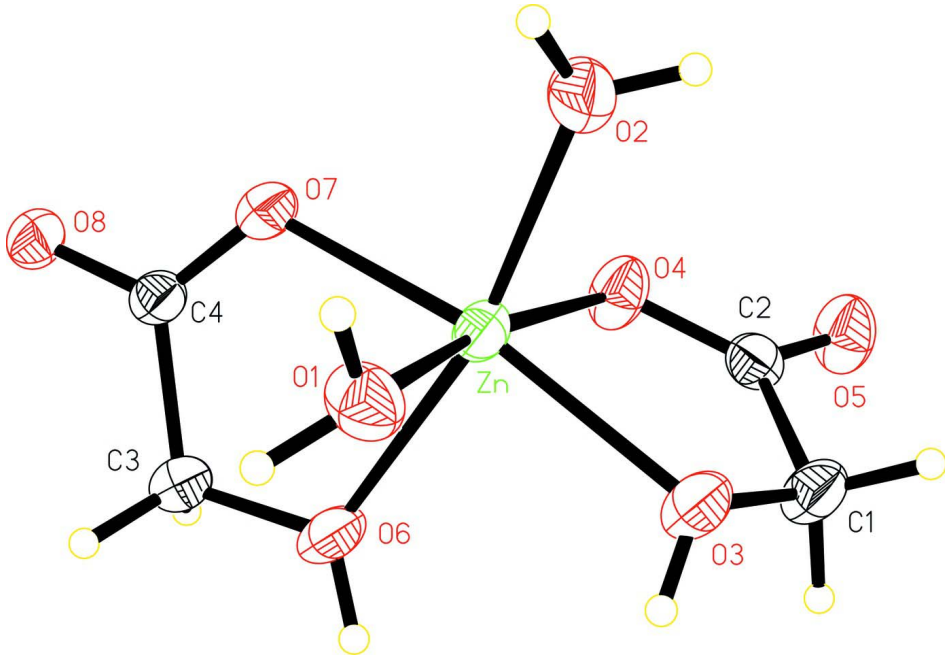

Figure 1

A view of the molecular structure of compound (I), showing the atom numbering scheme and dispacement ellipsods drawn at the 50% probability level.

cis-diaquadiglycolatozinc(II)

Crystal data

$[Zn(C_2H_3O_3)_2(H_2O)_2]$
$M_r = 251.49$
Monoclinic, $P2_1/c$
Hall symbol: -P 2ybc
$a = 11.391$ (2) Å
$b = 5.857$ (1) Å
$c = 12.511$ (2) Å
$\beta = 91.198$ (9)°
$V = 834.5$ (2) Å^3
$Z = 4$
$F(000) = 512$
$D_x = 2.002$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 20 reflections
$\theta = 9.6$–17.4°
$\mu = 2.96$ mm^{-1}
$T = 273$ K
Needle, colorless
0.3 × 0.3 × 0.1 mm

Data collection

Bruker P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$2\theta/\omega$ scans
Absorption correction: ψ scan (*SADABS*; Bruker, 2000)
$T_{min} = 0.425$, $T_{max} = 0.744$
2102 measured reflections
2102 independent reflections
1751 reflections with $I > 2\sigma(I)$
$R_{int} = 0.000$
$\theta_{max} = 28.5°$, $\theta_{min} = 3.3°$
$h = -15 \rightarrow 15$
$k = 0 \rightarrow 7$
$l = 0 \rightarrow 16$
3 standard reflections every 97 reflections
intensity decay: none

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.030$
$wR(F^2) = 0.072$
$S = 0.99$
2102 reflections
119 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0337P)^2 + 0.5879P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.030$
$\Delta\rho_{max} = 0.45$ e Å^{-3}
$\Delta\rho_{min} = -0.51$ e Å^{-3}
Extinction correction: *SHELXL97* (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Extinction coefficient: 0.0094 (8)

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*-factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn	0.76227 (2)	0.14144 (5)	0.65837 (2)	0.02431 (11)
O1	0.73409 (15)	0.0926 (3)	0.82160 (13)	0.0323 (4)
O2	0.91409 (16)	0.3091 (3)	0.69490 (15)	0.0349 (4)
O3	0.82248 (17)	−0.1945 (3)	0.64048 (13)	0.0307 (4)
O4	0.80776 (17)	0.1349 (3)	0.49645 (13)	0.0310 (4)
O5	0.90455 (17)	−0.0608 (4)	0.37503 (13)	0.0367 (4)
O6	0.58388 (16)	0.0302 (3)	0.63177 (15)	0.0339 (4)
O7	0.66840 (15)	0.4419 (3)	0.64627 (14)	0.0299 (4)
O8	0.49499 (15)	0.6096 (3)	0.62300 (13)	0.0275 (4)
C1	0.8665 (2)	−0.2479 (4)	0.53732 (19)	0.0297 (5)
H1A	0.8215	−0.3727	0.5062	0.036*
H1B	0.9477	−0.2968	0.5444	0.036*
C2	0.8588 (2)	−0.0423 (4)	0.46432 (18)	0.0242 (5)
C3	0.4989 (2)	0.2052 (4)	0.62244 (18)	0.0242 (5)
H2A	0.4422	0.1896	0.6787	0.029*
H2B	0.4575	0.1927	0.5542	0.029*
C4	0.5587 (2)	0.4364 (4)	0.63092 (16)	0.0218 (4)
H3	0.7697	−0.2865	0.6547	0.050*
H4	0.5587	−0.1048	0.6293	0.050*
H5	0.9803	0.2654	0.6719	0.050*
H6	0.9284	0.3898	0.7563	0.050*

H7	0.6612	0.0892	0.8427	0.050*
H8	0.7688	0.1759	0.8742	0.050*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn	0.02730 (16)	0.01890 (15)	0.02686 (15)	0.00221 (12)	0.00378 (10)	−0.00151 (11)
O1	0.0314 (9)	0.0381 (10)	0.0275 (8)	−0.0050 (8)	0.0044 (7)	−0.0032 (7)
O2	0.0280 (9)	0.0360 (10)	0.0408 (10)	−0.0002 (8)	0.0038 (7)	−0.0114 (8)
O3	0.0452 (10)	0.0204 (8)	0.0269 (8)	0.0026 (8)	0.0121 (7)	0.0011 (7)
O4	0.0404 (10)	0.0270 (9)	0.0260 (8)	0.0094 (8)	0.0063 (7)	0.0037 (7)
O5	0.0435 (11)	0.0390 (11)	0.0280 (9)	0.0100 (9)	0.0116 (8)	0.0036 (8)
O6	0.0327 (10)	0.0161 (8)	0.0527 (11)	0.0002 (8)	−0.0013 (8)	−0.0041 (8)
O7	0.0268 (9)	0.0174 (8)	0.0457 (10)	0.0012 (7)	0.0026 (7)	−0.0014 (8)
O8	0.0294 (9)	0.0198 (8)	0.0333 (9)	0.0036 (7)	0.0046 (7)	0.0029 (7)
C1	0.0414 (14)	0.0203 (11)	0.0277 (12)	0.0026 (11)	0.0100 (10)	−0.0006 (10)
C2	0.0216 (11)	0.0255 (12)	0.0257 (11)	−0.0017 (10)	0.0017 (8)	0.0014 (9)
C3	0.0294 (12)	0.0196 (10)	0.0238 (10)	−0.0008 (10)	−0.0008 (9)	−0.0002 (9)
C4	0.0284 (11)	0.0185 (10)	0.0188 (9)	−0.0011 (9)	0.0049 (8)	0.0010 (8)

Geometric parameters (Å, º)

Zn—O2	2.0325 (19)	O4—C2	1.259 (3)
Zn—O7	2.0630 (17)	O5—C2	1.247 (3)
Zn—O1	2.0935 (17)	O6—C3	1.412 (3)
Zn—O3	2.0974 (18)	O6—H4	0.8417
Zn—O4	2.1019 (17)	O7—C4	1.261 (3)
Zn—O6	2.1531 (19)	O8—C4	1.250 (3)
O1—H7	0.8768	C1—C2	1.513 (3)
O1—H8	0.9037	C1—H1A	0.9700
O2—H5	0.8522	C1—H1B	0.9700
O2—H6	0.9135	C3—C4	1.518 (3)
O3—C1	1.429 (3)	C3—H2A	0.9700
O3—H3	0.8293	C3—H2B	0.9700
O2—Zn—O7	92.38 (7)	C2—O4—Zn	116.58 (15)
O2—Zn—O1	89.65 (7)	C3—O6—Zn	115.85 (14)
O7—Zn—O1	95.65 (7)	C3—O6—H4	116.5
O2—Zn—O3	101.47 (8)	Zn—O6—H4	127.6
O7—Zn—O3	164.27 (7)	C4—O7—Zn	120.00 (16)
O1—Zn—O3	91.89 (7)	O3—C1—C2	110.72 (19)
O2—Zn—O4	89.99 (7)	O3—C1—H1A	109.5
O7—Zn—O4	94.74 (7)	C2—C1—H1A	109.5
O1—Zn—O4	169.61 (7)	O3—C1—H1B	109.5
O3—Zn—O4	78.01 (7)	C2—C1—H1B	109.5
O2—Zn—O6	167.62 (7)	H1A—C1—H1B	108.1
O7—Zn—O6	76.15 (7)	O5—C2—O4	124.2 (2)
O1—Zn—O6	86.90 (7)	O5—C2—C1	116.8 (2)

O3—Zn—O6	90.53 (7)	O4—C2—C1	119.00 (19)
O4—Zn—O6	95.53 (7)	O6—C3—C4	109.64 (19)
Zn—O1—H7	117.5	O6—C3—H2A	109.7
Zn—O1—H8	124.3	C4—C3—H2A	109.7
H7—O1—H8	101.3	O6—C3—H2B	109.7
Zn—O2—H5	122.2	C4—C3—H2B	109.7
Zn—O2—H6	125.1	H2A—C3—H2B	108.2
H5—O2—H6	107.2	O8—C4—O7	124.3 (2)
C1—O3—Zn	115.05 (14)	O8—C4—C3	117.4 (2)
C1—O3—H3	108.8	O7—C4—C3	118.3 (2)
Zn—O3—H3	110.3		

O2—Zn—O3—C1	−84.29 (19)	O2—Zn—O7—C4	−174.79 (18)
O7—Zn—O3—C1	67.0 (3)	O1—Zn—O7—C4	−84.91 (18)
O1—Zn—O3—C1	−174.31 (18)	O3—Zn—O7—C4	33.4 (4)
O4—Zn—O3—C1	3.24 (18)	O4—Zn—O7—C4	95.01 (18)
O6—Zn—O3—C1	98.77 (18)	O6—Zn—O7—C4	0.47 (17)
O2—Zn—O4—C2	94.74 (19)	Zn—O3—C1—C2	0.1 (3)
O7—Zn—O4—C2	−172.86 (19)	Zn—O4—C2—O5	−170.4 (2)
O1—Zn—O4—C2	6.7 (5)	Zn—O4—C2—C1	9.4 (3)
O3—Zn—O4—C2	−6.98 (18)	O3—C1—C2—O5	173.5 (2)
O6—Zn—O4—C2	−96.36 (19)	O3—C1—C2—O4	−6.3 (3)
O2—Zn—O6—C3	21.4 (5)	Zn—O6—C3—C4	1.6 (2)
O7—Zn—O6—C3	−1.22 (16)	Zn—O7—C4—O8	179.73 (17)
O1—Zn—O6—C3	95.41 (17)	Zn—O7—C4—C3	0.3 (3)
O3—Zn—O6—C3	−172.75 (16)	O6—C3—C4—O8	179.2 (2)
O4—Zn—O6—C3	−94.73 (17)	O6—C3—C4—O7	−1.3 (3)

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1—H8···O4^{i}	0.90	1.93	2.821 (3)	167
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