Diaquabis(4-formylbenzoato-κO)zinc(II)

Zhao-Peng Deng,[a] Shan Gao,[a] Li-Hua Huo[a] and Seik Weng Ng[b]*

[a]School of Chemistry and Materials Science, Heilongjiang University, Harbin 150080, People's Republic of China, and [b]Department of Chemistry, University of Malaya, 50603 Kuala Lumpur, Malaysia
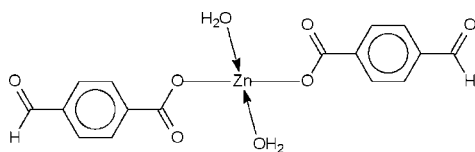
Correspondence e-mail: seikweng@um.edu.my

The Zn^{II} atom in the title compound, $[Zn(C_8H_5O_3)_2(H_2O)_2]$, which lies on a twofold rotation axis, is coordinated by two monodentate carboxylate groups and two water molecules in a tetrahedral geometry; the geometry is distorted towards octahedral owing to two long $Zn\cdots O_{carbonyl}$ contacts [2.512 (2) Å]. Hydrogen-bonding interactions give rise to a three-dimensional network. The formyl group is disordered approximately equally over two positions.

Related literature

A pseudo-polymorph of the title compound containing a solvent water molecule exists in a $P2/c$ modification, which features zinc in an unambiguous tetrahedral coordination geometry; see Deng et al. (2006).

Experimental

Crystal data

$[Zn(C_8H_5O_3)_2(H_2O)_2]$	$V = 1565.4$ (1) Å^3
$M_r = 399.64$	$Z = 4$
Monoclinic, $C2/c$	Mo $K\alpha$ radiation
$a = 27.537$ (1) Å	$\mu = 1.61$ mm^{-1}
$b = 5.0039$ (2) Å	$T = 295$ (2) K
$c = 12.0930$ (6) Å	$0.34 \times 0.26 \times 0.18$ mm
$\beta = 110.039$ (2)°	

Data collection

Rigaku R-AXIS RAPID diffractometer	7203 measured reflections
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	1785 independent reflections
$T_{min} = 0.532$, $T_{max} = 0.760$	1448 reflections with $I > 2\sigma(I)$
	$R_{int} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.082$	
$S = 1.09$	$\Delta\rho_{max} = 0.59$ e Å^{-3}
1785 reflections	$\Delta\rho_{min} = -0.38$ e Å^{-3}
132 parameters	
3 restraints	

Table 1
Selected bond lengths (Å).

Zn1—O1W	1.983 (2)	Zn1—O2	2.512 (2)
Zn1—O1	2.005 (2)		

Table 2
Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W1···O1^i	0.84 (1)	1.93 (1)	2.761 (2)	174 (3)
O1W—H1W2···O2^{ii}	0.84 (1)	1.88 (1)	2.720 (2)	174 (3)

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

Data collection: RAPID-AUTO (Rigaku, 1998); cell refinement: RAPID-AUTO; data reduction: CrystalStructure (Rigaku/MSC, 2002); program(s) used to solve structure: SHELXS97 (Sheldrick, 2008); program(s) used to refine structure: SHELXL97 (Sheldrick, 2008); molecular graphics: X-SEED (Barbour, 2001); software used to prepare material for publication: publCIF (Westrip, 2008).

References

Barbour, L. J. (2001). J. Supramol. Chem. **1**, 189–191.

Deng, Z.-P., Gao, S., Huo, L.-H. & Zhao, H. (2006). Acta Cryst. E**62**, m3524–m3526.

Higashi, T. (1995). ABSCOR. Rigaku Corporation, Tokyo, Japan.

Rigaku (1998). RAPID-AUTO. Rigaku Corporation, Tokyo, Japan.

Rigaku/MSC (2002). CrystalStructure. Rigaku/MSC, The Woodlands, Texas, USA.

Sheldrick, G. M. (2008). Acta Cryst. A**64**, 112–122.

Westrip, S. P. (2008). publCIF. In preparation.

supplementary materials

Diaquabis(4-formylbenzoato-*κO*)zinc(II)

Z.-P. Deng, S. Gao, L.-H. Huo and S. W. Ng

Comment

A pseuodpolymorph of the title compound containing a solvent water molecule was isolated from the reaction of zinc acetate and 4-formylbenzoic acid in the presence of sodium hydroxide (Deng *et al.*, 2006). The reaction with pyridine in place of sodium hydroxide yielded the title polymorph.

Experimental

Zinc diacetate dihydrate (2.2 g, 10 mmol) was added to anaqueous solution of 4-formylbenzoic acid (3.0 g, 20 mmol) that has earlier been treated with 1 ml pyridine to give a pH of 6. The solution was allowed to evaporate at room temperature; colorless prismatic crystals separated from the filtered solution after several days. C&N elemental analysis. Calc. $C_{16}H_{14}O_8Zn$: C 48.08, H3.53%. Found: C 48.06, H 3.56%.

Refinement

The formyl group is disordered over two sites; the ratio of the site occupation factors refined to a 0.508 (5):0.492 (5) ratio.

The carbon-bound H atoms were placed in calculated positions [C–H 0.93 Å and $U_{iso}(H)$ 1.2$U_{eq}(C)$], and were included in the refinement in the riding-model approximation. The water H-atoms were located in a difference Fourier map, and were refined with distance restraints of O–H 0.85±0.01 Å and H···H 1.39±0.01 Å; their displacement parameters were freely refined.

Figures
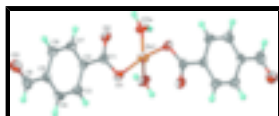
Fig. 1. Anisotropic displacement parameter plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radius.

Diaquabis(4-formylbenzoato-κO)zinc(II)

Crystal data

[Zn(C$_8$H$_5$O$_3$)$_2$(H$_2$O)$_2$]	$F_{000} = 816$
$M_r = 399.64$	$D_x = 1.696$ Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *Kα* radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 5776 reflections
$a = 27.537$ (1) Å	θ = 3.2–27.5º

$b = 5.0039\ (2)$ Å

$c = 12.0930\ (6)$ Å

$\beta = 110.039\ (2)°$

$V = 1565.4\ (1)$ Å^3

$Z = 4$

$\mu = 1.61$ mm^{-1}

$T = 295\ (2)$ K

Block, colorless

$0.34 \times 0.26 \times 0.18$ mm

Data collection

Rigaku R-AXIS RAPID diffractometer	1785 independent reflections
Radiation source: fine-focus sealed tube	1448 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.034$
Detector resolution: 10.000 pixels mm^{-1}	$\theta_{max} = 27.5°$
$T = 295(2)$ K	$\theta_{min} = 3.2°$
ω scans	$h = -35 \rightarrow 35$
Absorption correction: multi-scan (ABSCOR; Higashi, 1995)	$k = -6 \rightarrow 6$
$T_{min} = 0.532$, $T_{max} = 0.760$	$l = -15 \rightarrow 15$
7203 measured reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.029$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.082$	$w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.5739P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
1785 reflections	$\Delta\rho_{max} = 0.59$ e Å^{-3}
132 parameters	$\Delta\rho_{min} = -0.38$ e Å^{-3}
3 restraints	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	$U_{iso}*/U_{eq}$	Occ. (<1)
Zn1	0.5000	0.07300 (7)	0.7500	0.03500 (15)	
O1W	0.52887 (7)	−0.2045 (3)	0.67306 (15)	0.0420 (4)	
O1	0.44080 (6)	0.3312 (3)	0.69827 (13)	0.0347 (4)	
O2	0.46297 (6)	0.2522 (3)	0.54394 (14)	0.0396 (4)	
O3	0.28940 (13)	1.2782 (7)	0.2481 (3)	0.0537 (13)	0.508 (5)
O3'	0.25060 (16)	1.2320 (10)	0.3707 (4)	0.0757 (18)	0.492 (5)
H1W1	0.5398 (10)	−0.346 (3)	0.7104 (19)	0.051 (8)*	
H1W2	0.5313 (11)	−0.208 (5)	0.6055 (12)	0.062 (9)*	
C1	0.43534 (8)	0.3714 (4)	0.59025 (19)	0.0299 (5)	

C2	0.39595 (8)	0.5736 (4)	0.52428 (18)	0.0283 (4)	
C3	0.35743 (9)	0.6552 (5)	0.5675 (2)	0.0396 (5)	
H3	0.3554	0.5809	0.6363	0.048*	
C4	0.32199 (9)	0.8489 (6)	0.5070 (2)	0.0444 (6)	
H4	0.2963	0.9046	0.5357	0.053*	
C5	0.32469 (8)	0.9588 (4)	0.4047 (2)	0.0348 (5)	
C6	0.36283 (9)	0.8759 (5)	0.3612 (2)	0.0373 (5)	
H6	0.3645	0.9485	0.2919	0.045*	
C7	0.39830 (9)	0.6846 (5)	0.4219 (2)	0.0358 (5)	
H7	0.4240	0.6303	0.3932	0.043*	
C8	0.28767 (9)	1.1697 (5)	0.3399 (2)	0.0460 (6)	
H8	0.2621	1.2233	0.3691	0.055*	0.508 (5)
H8'	0.2924	1.2551	0.2760	0.055*	0.492 (5)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0384 (2)	0.02089 (19)	0.0487 (3)	0.000	0.01878 (17)	0.000
O1W	0.0676 (11)	0.0278 (8)	0.0359 (10)	0.0119 (8)	0.0247 (8)	0.0052 (7)
O1	0.0445 (9)	0.0313 (8)	0.0266 (8)	−0.0005 (7)	0.0101 (6)	0.0054 (6)
O2	0.0460 (9)	0.0351 (8)	0.0355 (9)	0.0121 (7)	0.0111 (7)	−0.0003 (7)
O3	0.051 (2)	0.055 (2)	0.052 (2)	0.0156 (17)	0.0141 (17)	0.0215 (19)
O3'	0.058 (3)	0.086 (4)	0.079 (3)	0.036 (2)	0.019 (2)	0.008 (3)
C1	0.0335 (11)	0.0244 (10)	0.0276 (11)	−0.0051 (8)	0.0051 (8)	−0.0011 (8)
C2	0.0318 (10)	0.0251 (10)	0.0264 (10)	−0.0022 (8)	0.0078 (8)	−0.0022 (9)
C3	0.0413 (12)	0.0478 (13)	0.0338 (13)	0.0033 (11)	0.0180 (10)	0.0055 (10)
C4	0.0356 (12)	0.0557 (15)	0.0458 (15)	0.0093 (11)	0.0190 (10)	−0.0025 (12)
C5	0.0314 (11)	0.0341 (12)	0.0337 (12)	0.0016 (9)	0.0044 (9)	−0.0032 (10)
C6	0.0418 (12)	0.0386 (12)	0.0322 (12)	0.0061 (10)	0.0135 (9)	0.0093 (10)
C7	0.0417 (12)	0.0366 (11)	0.0342 (12)	0.0106 (10)	0.0196 (10)	0.0037 (10)
C8	0.0368 (13)	0.0432 (13)	0.0497 (16)	0.0091 (11)	0.0043 (11)	−0.0036 (12)

Geometric parameters (Å, °)

Zn1—O1w	1.983 (2)	C2—C3	1.395 (3)
Zn1—O1w^i	1.983 (2)	C3—C4	1.392 (4)
Zn1—O1^i	2.005 (2)	C3—H3	0.9300
Zn1—O1	2.005 (2)	C4—C5	1.379 (4)
Zn1—O2	2.512 (2)	C4—H4	0.9300
O1W—H1W1	0.84 (1)	C5—C6	1.389 (3)
O1W—H1W2	0.84 (1)	C5—C8	1.490 (3)
O1—C1	1.279 (3)	C6—C7	1.385 (3)
O2—C1	1.241 (3)	C6—H6	0.9300
O3—C8	1.251 (4)	C7—H7	0.9300
O3'—C8	1.240 (5)	C8—H8	0.9300
C1—C2	1.498 (3)	C8—H8'	0.9300
C2—C7	1.379 (3)		
O1W—Zn1—O1W^i	91.11 (10)	C5—C4—H4	119.8

O1W—Zn1—O1[i]	100.59 (7)	C3—C4—H4	119.8
O1W[i]—Zn1—O1[i]	135.95 (7)	C4—C5—C6	120.0 (2)
O1W—Zn1—O1	135.95 (7)	C4—C5—C8	121.1 (2)
O1W[i]—Zn1—O1	100.59 (7)	C6—C5—C8	118.9 (2)
O1[i]—Zn1—O1	99.75 (9)	C7—C6—C5	119.6 (2)
Zn1—O1W—H1W1	117.8 (17)	C7—C6—H6	120.2
Zn1—O1W—H1W2	130.4 (17)	C5—C6—H6	120.2
H1W1—O1W—H1W2	111.8 (16)	C2—C7—C6	120.8 (2)
C1—O1—Zn1	102.62 (13)	C2—C7—H7	119.6
O2—C1—O1	120.65 (19)	C6—C7—H7	119.6
O2—C1—C2	121.9 (2)	O3—C8—O3'	116.3 (3)
O1—C1—C2	117.42 (19)	O3—C8—C5	123.1 (3)
C7—C2—C3	119.6 (2)	O3'—C8—C5	120.4 (3)
C7—C2—C1	120.14 (19)	O3—C8—H8	118.4
C3—C2—C1	120.2 (2)	C5—C8—H8	118.4
C4—C3—C2	119.5 (2)	O3'—C8—H8'	119.8
C4—C3—H3	120.2	C5—C8—H8'	119.8
C2—C3—H3	120.2	H8—C8—H8'	121.5
C5—C4—C3	120.4 (2)		
O1W—Zn1—O1—C1	−27.75 (17)	C3—C4—C5—C6	0.1 (4)
O1W[i]—Zn1—O1—C1	−130.56 (13)	C3—C4—C5—C8	−178.9 (2)
O1[i]—Zn1—O1—C1	88.73 (13)	C4—C5—C6—C7	−0.6 (4)
Zn1—O1—C1—O2	2.0 (2)	C8—C5—C6—C7	178.5 (2)
Zn1—O1—C1—C2	−175.83 (14)	C3—C2—C7—C6	−0.1 (3)
O2—C1—C2—C7	−17.2 (3)	C1—C2—C7—C6	−178.6 (2)
O1—C1—C2—C7	160.7 (2)	C5—C6—C7—C2	0.6 (4)
O2—C1—C2—C3	164.4 (2)	C4—C5—C8—O3	178.6 (3)
O1—C1—C2—C3	−17.8 (3)	C6—C5—C8—O3	−0.5 (4)
C7—C2—C3—C4	−0.3 (4)	C4—C5—C8—O3'	−7.0 (5)
C1—C2—C3—C4	178.2 (2)	C6—C5—C8—O3'	173.9 (3)
C2—C3—C4—C5	0.3 (4)		

Symmetry codes: (i) −x+1, y, −z+3/2.

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
O1W—H1W1⋯O1[ii]	0.84 (1)	1.93 (1)	2.761 (2)	174 (3)
O1W—H1W2⋯O2[iii]	0.84 (1)	1.88 (1)	2.720 (2)	174 (3)

Symmetry codes: (ii) −x+1, y−1, −z+3/2; (iii) −x+1, −y, −z+1.

Fig. 1