

Diaquabis(4-formylbenzoato- κ O)zinc(II)

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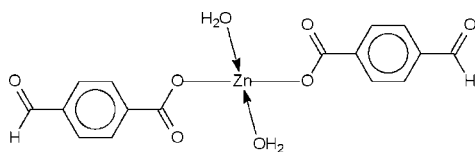
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Key indicators: single-crystal X-ray study; $T = 295$ K; mean $\sigma(\text{C}-\text{C}) = 0.003$ Å; disorder in main residue; R factor = 0.030; wR factor = 0.082; data-to-parameter ratio = 13.5.

The Zn^{II} atom in the title compound, $[\text{Zn}(\text{C}_8\text{H}_5\text{O}_3)_2(\text{H}_2\text{O})_2]$, 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

$[\text{Zn}(\text{C}_8\text{H}_5\text{O}_3)_2(\text{H}_2\text{O})_2]$

$M_r = 399.64$

Monoclinic, $C2/c$

$a = 27.537$ (1) Å

$b = 5.0039$ (2) Å

$c = 12.0930$ (6) Å

$\beta = 110.039$ (2)°

$V = 1565.4$ (1) Å³

$Z = 4$

Mo $K\alpha$ radiation

$\mu = 1.61$ mm⁻¹

$T = 295$ (2) K

$0.34 \times 0.26 \times 0.18$ mm

Data collection

Rigaku R-Axis RAPID

diffractometer

Absorption correction: multi-scan

(*ABSCOR*; Higashi, 1995)

$T_{\min} = 0.532$, $T_{\max} = 0.760$

7203 measured reflections

1785 independent reflections

1448 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.034$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.082$

$S = 1.09$

1785 reflections

132 parameters

3 restraints

H atoms treated by a mixture of independent and constrained refinement

$\Delta\rho_{\text{max}} = 0.59$ e Å⁻³

$\Delta\rho_{\text{min}} = -0.38$ e Å⁻³

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 \cdots O1 ⁱ	0.84 (1)	1.93 (1)	2.761 (2)	174 (3)
O1W—H1W2 \cdots 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/MS, 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: *pubCIF* (Westrip, 2008).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BT2669).

References

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supplementary materials

Acta Cryst. (2008). E64, m447 [doi:10.1107/S1600536808003152]

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Comment

A pseudopolymorph 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 an aqueous 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. C₁₆H₁₄O₈Zn: C 48.08, H 3.53%. 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 $U_{\text{iso}}(\text{H}) 1.2U_{\text{eq}}(\text{C})$], 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

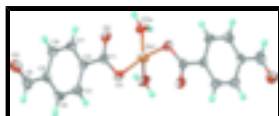


Fig. 1. Anisotropic displacement parameter plot of (I). Displacement ellipsoids are drawn at the 50% probability level and H atoms as spheres of arbitrary radius.

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Crystal data

[Zn(C₈H₅O₃)₂(H₂O)₂]

$M_r = 399.64$

Monoclinic, $C2/c$

Hall symbol: -C 2yc

$a = 27.537(1) \text{ \AA}$

$F_{000} = 816$

$D_x = 1.696 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 5776 reflections

$\theta = 3.2\text{--}27.5^\circ$

supplementary materials

$b = 5.0039 (2) \text{ \AA}$
 $c = 12.0930 (6) \text{ \AA}$
 $\beta = 110.039 (2)^\circ$
 $V = 1565.4 (1) \text{ \AA}^3$
 $Z = 4$

$\mu = 1.61 \text{ mm}^{-1}$
 $T = 295 (2) \text{ K}$
Block, colorless
 $0.34 \times 0.26 \times 0.18 \text{ mm}$

Data collection

Rigaku R-Axis RAPID
diffractometer
Radiation source: fine-focus sealed tube
Monochromator: graphite
Detector resolution: $10.000 \text{ pixels mm}^{-1}$
 $T = 295(2) \text{ K}$
 ω scans
Absorption correction: multi-scan
(ABSCOR; Higashi, 1995)
 $T_{\min} = 0.532, T_{\max} = 0.760$
7203 measured reflections

1785 independent reflections
1448 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.034$
 $\theta_{\max} = 27.5^\circ$
 $\theta_{\min} = 3.2^\circ$
 $h = -35 \rightarrow 35$
 $k = -6 \rightarrow 6$
 $l = -15 \rightarrow 15$

Refinement

Refinement on F^2
Least-squares matrix: full
 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.082$
 $S = 1.09$
1785 reflections
132 parameters
3 restraints
Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
 $w = 1/[\sigma^2(F_o^2) + (0.0444P)^2 + 0.5739P]$
where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.59 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.38 \text{ e \AA}^{-3}$
Extinction correction: none

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$	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 (\AA^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 (\AA , $^\circ$)

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

supplementary materials

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 ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O1W—H1W1 \cdots O1 ⁱⁱ	0.84 (1)	1.93 (1)	2.761 (2)	174 (3)
O1W—H1W2 \cdots 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

