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Aqua{4,4′-dimethoxy-2,2′-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato-κ^4O,O',N,N'}zinc(II)

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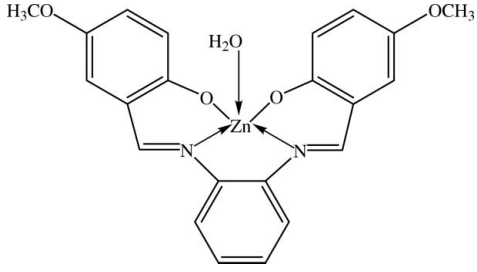
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.002 Å; R factor = 0.027; wR factor = 0.082; data-to-parameter ratio = 23.8.

In the title compound, $[Zn(C_{22}H_{18}N_2O_4)(H_2O)]$, the Zn^{II} center, on a crystallographic mirror plane, is in a five-coordinate N_2O_3 environment that is approximately square pyramidal, with the N_2O_2 tetradentate Schiff base ligand as the basal plane and the water molecule in the apical position. Intermolecular O—H···O and weak C—H···O interactions link the molecules into chains along [010]. These chains are further interconnected in a zigzag manner into a three-dimensional network.

Related literature

For related literature on values of bond lengths, see: Allen *et al.* (1987). For related structures, see, for example: Humphrey *et al.* (1999); Eltayeb, Teoh, Ng *et al.* (2007); Eltayeb, Teoh, Fun *et al.* (2007). For related literature, see: Tarafder *et al.* (2002).



Experimental

Crystal data

$[Zn(C_{22}H_{18}N_2O_4)(H_2O)]$
M_r = 457.79
Orthorhombic, $Pmn2_1$
a = 23.3617 (7) Å
b = 4.9121 (1) Å
c = 7.9431 (2) Å
V = 911.51 (4) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.39 mm^{-1}
T = 100.0 (1) K
0.38 × 0.23 × 0.10 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2005)
T_{min} = 0.623, T_{max} = 0.874
29716 measured reflections
3359 independent reflections
3179 reflections with $I > 2\sigma(I)$
R_{int} = 0.047

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.027
$wR(F^2)$ = 0.082
S = 1.16
3359 reflections
141 parameters
2 restraints
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.49 e Å^{-3}
$\Delta\rho_{min}$ = −0.70 e Å^{-3}
Absolute structure: Flack (1983), 1573 Friedel pairs
Flack parameter: 0.012 (10)

Table 1
Selected geometric parameters (Å, °).

Zn1—O1	1.9758 (12)	Zn1—N1	2.0825 (14)
Zn1—O1*W*	2.069 (2)		
O1—Zn1—O1^{i}	91.64 (7)	O1—Zn1—N1	90.52 (6)
O1—Zn1—O1*W*	104.52 (6)	O1*W*—Zn1—N1	96.16 (6)
C11—O2—C4—C5	−9.7 (2)	C11—O2—C4—C3	170.21 (16)

Symmetry code: (i) $-x, y, z$.

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1*W*—H1*W*1···O1^{ii}	0.82	1.96	2.711 (2)	153
C7—H7*A*···O2^{iii}	0.94	2.56	3.358 (2)	144

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *APEX2*; data reduction: *SAINT* (Bruker, 2005); program(s) used to solve structure: *SHELXTL* (Sheldrick, 1998); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2265).

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supplementary materials

Acta Cryst. (2007). E**63**, m1633-m1634 [**doi:10.1107/S1600536807022064**]

Aqua{4,4'-dimethoxy-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato-κ^4O,O',N,N'}zinc(II)

N. E. Eltayeb, S. G. Teoh, S. Chantrapromma, H.-K. Fun and K. Ibrahim

Comment

It is well known that zinc complexes with Schiff-bases are active in biological systems and show very good activity against the leukemic cell (Tarafder et al., 2002). Recently, we reported the crystal structure of {2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato- κ^4O,N,N',O'}(pyridine-κN)zinc(II) (Eltayeb et al., 2007). Herein, we report the crystal structure of (I).

In (I), the Zn1 and O1W atoms lie on a mirror plane and the asymmetric unit therefore contains only half of the molecule. The tetradentate Schiff-base ligand is almost planar with a maximum deviation from the C1–C10/O1–O2/N1 plane of 0.053 (3) Å for atom N1. The methoxy group is slightly twisted from the C1–C10/O1–O2/N1 mean plane as indicated by the torsion angle C11/O2/C4/C5 of −9.6 (2)°. The ligand is coordinated to Zn1 as a tetradentate ligand via through two N and two O donor atoms, resulting in a square-pyramidal Zn^{II} center whose fifth (axial) position is occupied by a water molecule (Fig. 1). The four atoms N1, O1, $N1^{i}$ and $O1^{i}$ [i = -x, y, z] form the basal plane, and the Zn^{II} is displaced 0.044 (7) Å out of this mean basal plane towards the axial water molecule. Bond lengths and angles observed in the structure are normal (Allen et al., 1987).

In the crystal of (I) in Fig. 2, the water molecule is involved in an intermolecular O—H···O hydrogen bond [O1W—H1W···O1; symmetry code x, −1 + y, +z] and the ligand is involved in weak C—H···O intermolecular interaction [C7—H7A···O2; symmetry code 1/2 − x, −y, −1/2 + z] (Table 2). The molcecules are linked into chains along the b axis. These chains are further interconnected in a zigzag manner into a three-dimensional network.

Experimental

The title compound (I) was synthesized by adding 5-methoxy-2-hydroxybenzaldehyde (0.610 g, 4 mmol) to a solution of *o*-phenylenediamine (0.216 g, 2 mmol) in ethanol 95% (20 ml). The mixture was refluxed with stirring for half an hour. Zinc chloride (0.272 g, 2 mmol) in ethanol (10 ml) was then added, followed by triethylamine (0.5 ml,3.6 mmol). The mixture was stirred at room temperature for two h. An orange-red precipitate was obtained; this was washed by about 5 ml e thanol, dried, and then washed by copious amount of diethyl ether. This precipitate was then dissolved in 25 ml of pyridine. Red single crystals were formed after two months.

Refinement

The water hydrogen atoms were located in a difference map and were restrained with an O1W—H distance of 0.82 Å. All other H atoms were positioned geometrically and allowed to ride on their parent atoms with C—H distances in the range 0.93–0.96 Å. The U_{iso} values were constrained to be $1.5U_{eq}$ of the carrier atom for methyl H atoms and $1.2U_{eq}$ for the remaining H atoms. A rotating group model was used for the methyl groups; 1573 Friedel pairs were used to determine the absolute structure.

Figures

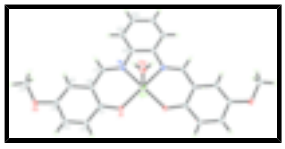

Fig. 1. The asymmetric unit of (I), showing 50% probability displacement ellipsoids and the atomic numbering.

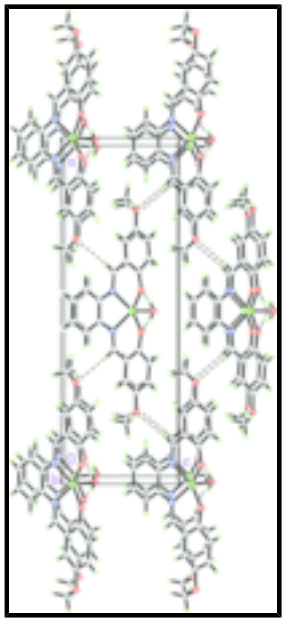

Fig. 2. The crystal packing of (I), viewed along the *b* axis. Hydrogen bonds are shown as dash lines.

Aqua{4,4'-dimethoxy-2,2'-[1,2-phenylenebis(nitrilomethylidyne)]diphenolato- κ^4O,O',N,N'}zinc(II)

Crystal data

$[Zn(C_{22}H_{18}N_2O_4)(H_2O)]$	F_{000} = 472
M_r = 457.79	D_x = 1.668 Mg m^{-3}
Orthorhombic, $Pmn2_1$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: P 2ac -2	Cell parameters from 3359 reflections
a = 23.3617 (7) Å	θ = 1.7–32.5º
b = 4.9121 (1) Å	μ = 1.39 mm^{-1}
c = 7.9431 (2) Å	T = 100.0 (1) K
V = 911.51 (4) Å^3	Plate, red
Z = 2	0.38 × 0.23 × 0.10 mm

Data collection

Bruker SMART APEX2 CCD area-detector diffractometer	3359 independent reflections
Radiation source: fine-focus sealed tube	3179 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.047
Detector resolution: 8.33 pixels mm^{-1}	θ_{max} = 32.5º

T = 100.0(1) K	θ_{min} = 1.7º
ω scans	h = −35→35
Absorption correction: multi-scan (SADABS; Bruker, 2005)	k = −7→7
T_{min} = 0.623, T_{max} = 0.874	l = −12→12
29716 measured reflections	

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.027	$w = 1/[\sigma^2(F_o^2) + (0.0476P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.082	$(\Delta/\sigma)_{max} < 0.001$
S = 1.16	$\Delta\rho_{max}$ = 0.49 e Å^{-3}
3359 reflections	$\Delta\rho_{min}$ = −0.70 e Å^{-3}
141 parameters	Extinction correction: none
2 restraints	Absolute structure: Flack (1983)
Primary atom site location: structure-invariant direct methods	Flack parameter: 0.012 (10)
Secondary atom site location: difference Fourier map	

Special details

Experimental. The low-temparture data was collected with the Oxford Cyrosystem Cobra low-temperature attachment.

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 (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Zn1	0.0000	0.05705 (5)	0.10449 (4)	0.01253 (7)
O1	0.06065 (5)	0.3095 (3)	0.17986 (17)	0.0153 (2)
O2	0.29634 (5)	0.2460 (3)	0.13415 (15)	0.0170 (3)
O1W	0.0000	−0.2503 (4)	0.2826 (2)	0.0169 (3)
H1W1	0.0270	−0.3532	0.2651	0.058 (11)*
C1	0.11623 (7)	0.2828 (4)	0.1586 (2)	0.0133 (3)
C2	0.15306 (8)	0.4597 (3)	0.2488 (2)	0.0146 (3)
H2A	0.1369	0.5913	0.3184	0.017*
C3	0.21158 (8)	0.4445 (3)	0.2376 (2)	0.0148 (3)

H3A	0.2343	0.5649	0.2985	0.018*
C4	0.23727 (7)	0.2473 (4)	0.1342 (2)	0.0143 (3)
C5	0.20328 (7)	0.0725 (3)	0.0424 (2)	0.0133 (3)
H5A	0.2203	−0.0567	−0.0270	0.016*
C6	0.14266 (7)	0.0866 (3)	0.0522 (2)	0.0128 (3)
C7	0.11221 (7)	−0.1066 (4)	−0.0513 (2)	0.0136 (3)
H7A	0.1340	−0.2240	−0.1170	0.016*
C8	0.03036 (6)	−0.3179 (3)	−0.1707 (2)	0.0127 (3)
C9	0.05973 (7)	−0.4942 (4)	−0.2795 (2)	0.0152 (3)
H9A	0.0995	−0.4962	−0.2795	0.018*
N1	0.05703 (6)	−0.1304 (3)	−0.06032 (18)	0.0128 (3)
C10	0.02998 (6)	−0.6654 (3)	−0.3870 (3)	0.0156 (3)
H10A	0.0499	−0.7806	−0.4593	0.019*
C11	0.32314 (8)	0.0220 (4)	0.0525 (3)	0.0185 (3)
H11A	0.3638	0.0333	0.0678	0.028*
H11B	0.3144	0.0266	−0.0655	0.028*
H11C	0.3093	−0.1451	0.1002	0.028*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.01071 (11)	0.01086 (11)	0.01601 (12)	0.000	0.000	−0.00136 (12)
O1	0.0101 (5)	0.0129 (5)	0.0231 (6)	−0.0003 (4)	−0.0004 (4)	−0.0032 (5)
O2	0.0102 (5)	0.0185 (6)	0.0223 (8)	−0.0005 (4)	−0.0013 (4)	−0.0038 (5)
O1W	0.0191 (8)	0.0129 (8)	0.0188 (8)	0.000	0.000	0.0001 (7)
C1	0.0123 (7)	0.0122 (7)	0.0154 (7)	0.0004 (5)	−0.0006 (5)	0.0012 (5)
C2	0.0133 (7)	0.0113 (7)	0.0191 (8)	0.0007 (5)	−0.0001 (6)	−0.0014 (6)
C3	0.0134 (7)	0.0130 (8)	0.0179 (8)	−0.0021 (5)	−0.0013 (6)	−0.0006 (6)
C4	0.0116 (6)	0.0137 (7)	0.0176 (10)	0.0004 (5)	−0.0010 (5)	0.0009 (5)
C5	0.0102 (6)	0.0136 (7)	0.0161 (7)	0.0003 (5)	0.0010 (5)	−0.0003 (6)
C6	0.0110 (7)	0.0129 (7)	0.0144 (6)	−0.0008 (5)	−0.0011 (5)	0.0005 (5)
C7	0.0122 (7)	0.0147 (7)	0.0140 (7)	−0.0005 (5)	0.0010 (6)	−0.0007 (5)
C8	0.0122 (7)	0.0118 (7)	0.0141 (7)	−0.0010 (5)	−0.0001 (5)	0.0001 (6)
C9	0.0118 (7)	0.0176 (7)	0.0161 (8)	0.0010 (6)	0.0004 (6)	−0.0017 (6)
N1	0.0122 (6)	0.0116 (6)	0.0147 (6)	−0.0009 (5)	−0.0006 (5)	−0.0014 (5)
C10	0.0148 (6)	0.0149 (6)	0.0170 (7)	0.0013 (5)	0.0014 (8)	−0.0040 (8)
C11	0.0133 (7)	0.0178 (8)	0.0244 (9)	0.0005 (6)	0.0002 (6)	−0.0018 (6)

Geometric parameters (Å, °)

Zn1—O1	1.9758 (12)	C5—C6	1.420 (2)
Zn1—O1^{i}	1.9758 (12)	C5—H5A	0.9300
Zn1—O1W	2.069 (2)	C6—C7	1.443 (2)
Zn1—N1	2.0825 (14)	C7—N1	1.296 (2)
Zn1—N1^{i}	2.0826 (14)	C7—H7A	0.9300
O1—C1	1.316 (2)	C8—C9	1.403 (2)
O2—C4	1.3800 (19)	C8—N1	1.416 (2)
O2—C11	1.422 (2)	C8—C8^{i}	1.419 (3)

O1W—H1W1	0.8200	C9—C10	1.385 (3)
C1—C2	1.417 (2)	C9—H9A	0.9300
C1—C6	1.423 (2)	C10—C10^{i}	1.401 (3)
C2—C3	1.372 (2)	C10—H10A	0.9300
C2—H2A	0.9300	C11—H11A	0.9600
C3—C4	1.404 (2)	C11—H11B	0.9600
C3—H3A	0.9300	C11—H11C	0.9600
C4—C5	1.379 (2)		
O1—Zn1—O1^{i}	91.64 (7)	C4—C5—H5A	119.5
O1—Zn1—O1W	104.52 (6)	C6—C5—H5A	119.5
O1^{i}—Zn1—O1W	104.52 (6)	C5—C6—C1	119.90 (15)
O1—Zn1—N1	90.52 (6)	C5—C6—C7	115.35 (15)
O1^{i}—Zn1—N1	157.94 (6)	C1—C6—C7	124.75 (16)
O1W—Zn1—N1	96.16 (6)	N1—C7—C6	125.52 (16)
O1—Zn1—N1^{i}	157.94 (6)	N1—C7—H7A	117.2
O1^{i}—Zn1—N1^{i}	90.52 (6)	C6—C7—H7A	117.2
O1W—Zn1—N1^{i}	96.16 (6)	C9—C8—N1	124.60 (14)
N1—Zn1—N1^{i}	79.56 (8)	C9—C8—C8^{i}	119.28 (10)
C1—O1—Zn1	127.32 (11)	N1—C8—C8^{i}	116.10 (8)
C4—O2—C11	116.36 (14)	C10—C9—C8	120.61 (15)
Zn1—O1W—H1W1	109.5	C10—C9—H9A	119.7
O1—C1—C2	118.24 (16)	C8—C9—H9A	119.7
O1—C1—C6	124.88 (16)	C7—N1—C8	122.04 (15)
C2—C1—C6	116.87 (15)	C7—N1—Zn1	124.16 (12)
C3—C2—C1	122.59 (16)	C8—N1—Zn1	113.29 (10)
C3—C2—H2A	118.7	C9—C10—C10^{i}	120.11 (10)
C1—C2—H2A	118.7	C9—C10—H10A	119.9
C2—C3—C4	120.10 (16)	C10^{i}—C10—H10A	119.9
C2—C3—H3A	119.9	O2—C11—H11A	109.5
C4—C3—H3A	119.9	O2—C11—H11B	109.5
C5—C4—O2	124.95 (15)	H11A—C11—H11B	109.5
C5—C4—C3	119.53 (15)	O2—C11—H11C	109.5
O2—C4—C3	115.52 (15)	H11A—C11—H11C	109.5
C4—C5—C6	120.99 (16)	H11B—C11—H11C	109.5
O1^{i}—Zn1—O1—C1	−174.80 (12)	C2—C1—C6—C7	−178.82 (16)
O1W—Zn1—O1—C1	79.74 (15)	C5—C6—C7—N1	179.40 (17)
N1—Zn1—O1—C1	−16.75 (15)	C1—C6—C7—N1	−0.8 (3)
N1^{i}—Zn1—O1—C1	−79.4 (2)	N1—C8—C9—C10	−178.03 (17)
Zn1—O1—C1—C2	−168.48 (12)	C8^{i}—C8—C9—C10	0.5 (2)
Zn1—O1—C1—C6	10.9 (3)	C6—C7—N1—C8	177.22 (16)
O1—C1—C2—C3	178.75 (17)	C6—C7—N1—Zn1	−11.6 (3)
C6—C1—C2—C3	−0.7 (3)	C9—C8—N1—C7	0.1 (3)
C1—C2—C3—C4	−0.3 (3)	C8^{i}—C8—N1—C7	−178.52 (13)
C11—O2—C4—C5	−9.7 (2)	C9—C8—N1—Zn1	−172.00 (14)
C11—O2—C4—C3	170.21 (16)	C8^{i}—C8—N1—Zn1	9.43 (12)

C2—C3—C4—C5	1.0 (3)	O1—Zn1—N1—C7	16.93 (15)
C2—C3—C4—O2	−178.87 (16)	O1^{i}—Zn1—N1—C7	112.60 (18)
O2—C4—C5—C6	179.13 (15)	O1W—Zn1—N1—C7	−87.74 (15)
C3—C4—C5—C6	−0.7 (3)	N1^{i}—Zn1—N1—C7	177.11 (12)
C4—C5—C6—C1	−0.3 (3)	O1—Zn1—N1—C8	−171.21 (12)
C4—C5—C6—C7	179.54 (16)	O1^{i}—Zn1—N1—C8	−75.55 (19)
O1—C1—C6—C5	−178.45 (16)	O1W—Zn1—N1—C8	84.12 (11)
C2—C1—C6—C5	1.0 (2)	N1^{i}—Zn1—N1—C8	−11.03 (14)
O1—C1—C6—C7	1.8 (3)	C8—C9—C10—C10^{i}	−0.5 (2)

Symmetry codes: (i) −*x*, *y*, *z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W1···O1^{ii}	0.82	1.96	2.711 (2)	153
C7—H7A···O2^{iii}	0.94	2.56	3.358 (2)	144

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

Fig. 1

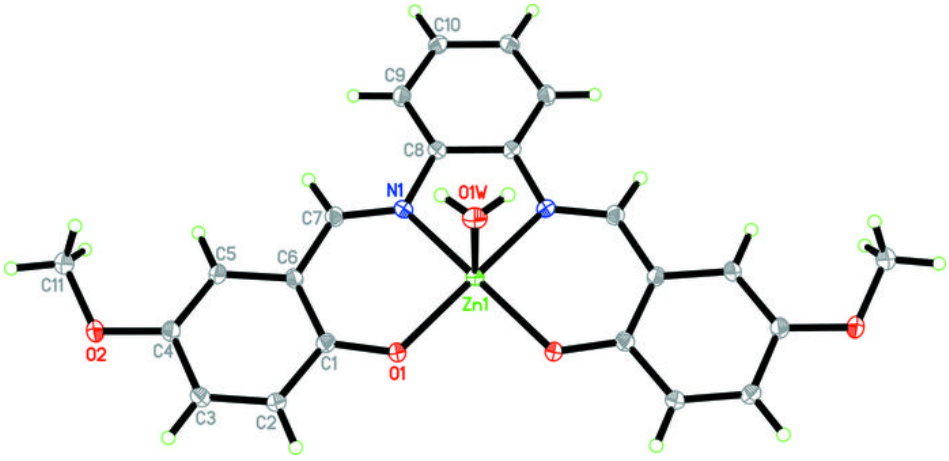

Fig. 2