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Aqua{6,6′-dimethoxy-2,2′-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-κ^4O,N,N',O'}cobalt(II)

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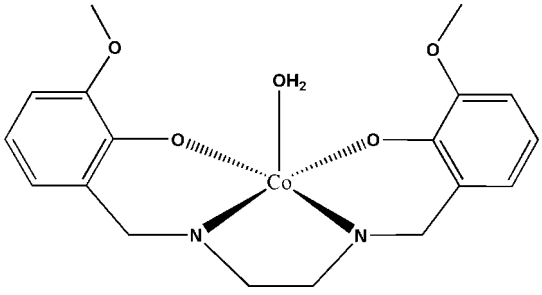
Key indicators: single-crystal X-ray study; T = 173 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.037; wR factor = 0.101; data-to-parameter ratio = 12.6.

In the title compound, $[Co(C_{18}H_{18}N_2O_4)(H_2O)]$, the Co^{II} atom is coordinated in a distorted square-pyramidal geometry defined by two O atoms and two N atoms in the basal positions, and one water molecule in the apical position. The Co^{II} atom and water O atom lie on a symmetry plane defining C_s molecular symmetry. In the crystal structure, the ethylenediamine group is disordered over two positions with equal occupancy. Molecules are linked into chains *via* O—H···O hydrogen bonds.

Related literature

For related literature, see: Guo *et al.* (2002).



Experimental

Crystal data

$[Co(C_{18}H_{18}N_2O_4)(H_2O)]$
M_r = 403.29
Orthorhombic, *Pnma*
a = 8.9827 (6) Å
b = 24.8632 (16) Å
c = 7.5784 (5) Å
V = 1692.55 (19) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.05 mm^{-1}
T = 173 (2) K
0.49 × 0.46 × 0.19 mm

Data collection

Bruker SMART 1000 CCD diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.628, T_{max} = 0.826
6807 measured reflections
1694 independent reflections
1389 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.037
$wR(F^2)$ = 0.101
S = 1.07
1694 reflections
134 parameters
4 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.76 e Å^{-3}
$\Delta\rho_{min}$ = −0.39 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Co1—O1	1.979 (2)	Co1—O1*W*	2.066 (3)
Co1—N1	2.036 (3)		
O1^i—Co1—O1	91.99 (12)	O1—Co1—O1*W*	106.80 (8)
O1—Co1—N1	89.55 (10)	N1—Co1—O1*W*	98.41 (13)
N1^i—Co1—N1	77.4 (2)		

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

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

D—H···A	D—H	H···A	D···A	D—H···A
O1*W*—H1*W*···O1^{ii}	0.848 (10)	2.100 (18)	2.830 (3)	144.0 (17)
O1*W*—H1*W*···O2^{ii}	0.848 (10)	2.242 (10)	2.962 (2)	142.7 (19)

Symmetry code: (ii) $x-\frac{1}{2}, -y+\frac{1}{2}, -z+\frac{5}{2}$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT-Plus* (Bruker, 2003); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2005); 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: KP2129).

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supplementary materials

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Aqua{6,6'-dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato-κ^4O,N,N',O'}cobalt(II)

G.-B. Jiang, S.-H. Zhang and M.-H. Zeng

Comment

Using H_2L [where H_2L is *N*,*N*'-ethylene-bis(3-methoxysalicylaldiminato)], we have hydrothermally prepared the title compound, (I), namely Co(*L*)(H_2O). As an example of penta coordinated Co(II) complexes there are series of complexes with 1,4-diazacycloheptane (DACH) functionalized by additional imidazole or pyridine donor pendants [CoL1Cl](ClO4)·H2O, [CoL2Cl](ClO4) and [CoL3Cl](ClO4)·CH3OH, where L1=1,4-bis(imidazole-4-ylmethyl)-DACH, L2=1,4-bis(*N*-1-methyl-imidazol-2-ylmethyl)-DACH and L3=1,4-bis(pyridyl-2-ylmethyl)-DACH that were synthesized and characterized (Guo *et al.*, 2002). In the present structure Co(II) atom is coordinated by two phenolato oxygen atoms and two imine nitrogen atoms from L^{2-} ligand, and one water molecule to furnish a distorted square pyramidal coordination geometry (Fig. 1, Table 1). The O atom of water molecule is at the apical position of square pyramid and the other two O atoms and two N atoms are at the base of square pyramid. The Co^{II} and O atom of water molecule lie on a symmetry plane. The two C atoms of ethylenediamine moiety are disordered over two positions with equal occupancy. The complex generates 1-D chain through hydrogen bonds between water molecule and phenolato oxygen (Table 2 and Fig. 2).

Experimental

2-Hydroxy-3-methoxy-benzaldehyde (0.152 g, 1 mmol), ethane-1,2-diamine (0.120 g, 2 mmol) and $Co(NO_3)_2·6H_2O$ (0.290 g, 1 mmol) were dissolved in a mixture solution (8 ml of methanol: acetonitrile = 1:1(v / v)). The solution was sealed in a 15 ml Teflon-lined stainless steel bomb and held at 353 K for 5 d. Then, a bomb was cooled to room temperature and red block crystals were filtered off, washed with methanol and dried at room temperature. Elemental analysis, calcd (%) for $C_{18}H_{20}CoN_2O_5$: C 62.78, H 5.85, N 8.13; found (%): C 62.56, H 5.92, N 8.11.

Refinement

There is positional disorder of C9 and $C9'^{i}$ over two equally occupied sites. It was assumed that the ethylenediamine moiety has two possible conformations, namely N1–C9(H9A/H9B)–$C9'^{i}$($H9C^{i}$/$H9D^{i}$)–$N1'^{i}$ and N1'–C9'(H9C/H9D)–$C9^{i}$($H9A^{i}$/$H9B^{i}$)–$N1^{i}$[symmetry code: (i) *x*, 1/2 − *y*, *z*]. H atoms of the water molecule were located in a difference Fourier map, but their distances and angles were restrained to literature values with U(H) = 1.5 times U_{eq}(O). All other H atoms were positioned geometrically and refined as riding atoms, with C–H distances of 0.95–0.98 Å and U(H) = 1.2–1.5 times U_{eq}(C).

Figures

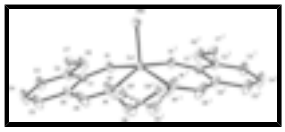

Fig. 1. The molecular structure of (I) with atom labels and the 50% probability displacement ellipsoids for non-H atoms. One of the disorder part is shown by a dashed line. The molecular symmetry C_s is generated by symmetry code: (i) *x*, 1/2 − *y*, *z*.

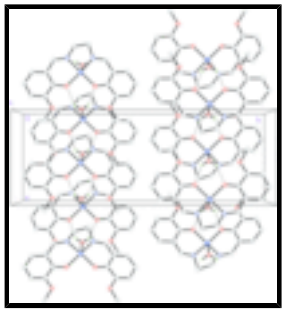

Fig. 2. The packing of (I), showing two chains of molecules connected by O—H···O hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

Aqua{6,6'-dimethoxy-2,2'-[ethane-1,2-diylbis(nitrilomethylidyne)]diphenolato- κ^4O,N,N',O'}cobalt(II)

Crystal data

$[Co(C_{18}H_{18}N_2O_4)(H_2O_1)]$	F_{000} = 836
M_r = 403.29	D_x = 1.583 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 3183 reflections
a = 8.9827 (6) Å	θ = 2.8–28.1º
b = 24.8632 (16) Å	μ = 1.05 mm^{-1}
c = 7.5784 (5) Å	*T* = 173 (2) K
V = 1692.55 (19) Å^3	Block, red
Z = 4	0.49 × 0.46 × 0.19 mm

Data collection

Bruker SMART 1000 CCD diffractometer	1694 independent reflections
Radiation source: fine-focus sealed tube	1389 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.027
T = 173(2) K	θ_{max} = 26.0º
ω scans	θ_{min} = 2.8º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −10→11
T_{min} = 0.628, T_{max} = 0.826	*k* = −30→20
6807 measured reflections	*l* = −9→7

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.101$

$S = 1.07$

1694 reflections

134 parameters

4 restraints

Primary atom site location: structure-invariant direct methods

Hydrogen site location: inferred from neighbouring sites

H atoms treated by a mixture of independent and constrained refinement

$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 2.1284P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.001$

$\Delta\rho_{max} = 0.76$ e Å^{-3}

$\Delta\rho_{min} = -0.39$ e Å^{-3}

Extinction correction: none

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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Co1	0.05985 (6)	0.2500	1.06224 (7)	0.0225 (2)	
C1	0.2009 (3)	0.35745 (12)	1.0129 (4)	0.0270 (6)	
C2	0.3278 (4)	0.39125 (13)	1.0321 (4)	0.0330 (7)	
C3	0.3227 (4)	0.44530 (14)	0.9907 (6)	0.0467 (9)	
H3	0.4080	0.4672	1.0091	0.056*	
C4	0.1929 (5)	0.46784 (15)	0.9220 (6)	0.0554 (11)	
H4	0.1895	0.5050	0.8934	0.066*	
C5	0.0714 (4)	0.43626 (15)	0.8964 (6)	0.0500 (10)	
H5	−0.0162	0.4517	0.8478	0.060*	
C6	0.0718 (3)	0.38107 (13)	0.9399 (4)	0.0344 (7)	
C7	0.5866 (4)	0.39539 (17)	1.1006 (6)	0.0544 (11)	
H7A	0.6086	0.4095	0.9827	0.082*	
H7B	0.6679	0.3719	1.1390	0.082*	
H7C	0.5763	0.4254	1.1837	0.082*	
C8	−0.0616 (4)	0.35127 (16)	0.9037 (5)	0.0502 (11)	
H8	−0.1407	0.3701	0.8480	0.060*	
C9	−0.2007 (7)	0.2696 (3)	0.8386 (10)	0.0364 (16)	0.50
H9A	−0.1631	0.2612	0.7233	0.044*	0.50
H9B	−0.2889	0.2910	0.8247	0.044*	0.50
C9'	−0.2370 (6)	0.2830 (3)	0.9378 (10)	0.0315 (14)	0.50

H9C	−0.3015	0.3063	0.8717	0.038*	0.50
H9D	−0.2767	0.2768	1.0537	0.038*	0.50
N1	−0.0838 (3)	0.30121 (14)	0.9399 (5)	0.0623 (11)	0.50
N1'	−0.0838 (3)	0.30121 (14)	0.9399 (5)	0.0623 (11)	0.50
O1	0.2129 (2)	0.30725 (8)	1.0632 (3)	0.0276 (5)	
O2	0.4510 (2)	0.36547 (10)	1.0954 (3)	0.0431 (6)	
O1W	−0.0369 (3)	0.2500	1.3095 (4)	0.0244 (6)	
H1W	−0.082 (3)	0.2221 (2)	1.346 (4)	0.037*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0178 (3)	0.0214 (3)	0.0284 (3)	0.000	−0.0003 (2)	0.000
C1	0.0252 (15)	0.0241 (15)	0.0317 (15)	−0.0012 (12)	0.0006 (12)	0.0019 (12)
C2	0.0308 (17)	0.0279 (16)	0.0402 (18)	−0.0021 (14)	−0.0052 (14)	0.0066 (14)
C3	0.044 (2)	0.0273 (18)	0.069 (2)	−0.0093 (16)	−0.0113 (19)	0.0082 (17)
C4	0.054 (2)	0.0252 (18)	0.087 (3)	−0.0007 (17)	−0.008 (2)	0.020 (2)
C5	0.039 (2)	0.0367 (19)	0.074 (3)	0.0063 (16)	−0.0083 (19)	0.0234 (19)
C6	0.0291 (16)	0.0326 (17)	0.0416 (18)	0.0003 (14)	−0.0011 (15)	0.0117 (14)
C7	0.036 (2)	0.058 (2)	0.070 (3)	−0.0231 (18)	−0.0205 (18)	0.030 (2)
C8	0.0273 (17)	0.052 (2)	0.072 (3)	−0.0039 (16)	−0.0160 (17)	0.037 (2)
C9	0.024 (3)	0.040 (4)	0.045 (4)	0.006 (3)	−0.008 (3)	0.010 (3)
C9'	0.016 (3)	0.038 (4)	0.040 (4)	0.006 (3)	0.003 (3)	0.006 (3)
N1	0.0272 (16)	0.060 (2)	0.100 (3)	−0.0151 (15)	−0.0277 (17)	0.051 (2)
N1'	0.0272 (16)	0.060 (2)	0.100 (3)	−0.0151 (15)	−0.0277 (17)	0.051 (2)
O1	0.0208 (10)	0.0209 (10)	0.0412 (12)	−0.0009 (8)	−0.0020 (9)	0.0054 (9)
O2	0.0276 (12)	0.0335 (12)	0.0682 (17)	−0.0103 (10)	−0.0141 (11)	0.0174 (12)
O1W	0.0225 (14)	0.0217 (14)	0.0291 (15)	0.000	0.0029 (12)	0.000

Geometric parameters (Å, °)

Co1—O1^{i}	1.979 (2)	C5—H5	0.9500
Co1—O1	1.979 (2)	C6—C8	1.435 (5)
Co1—N1^{i}	2.036 (3)	C7—O2	1.427 (4)
Co1—N1	2.036 (3)	C7—H7A	0.9800
Co1—O1W	2.066 (3)	C7—H7B	0.9800
C1—O1	1.309 (3)	C7—H7C	0.9800
C1—C6	1.413 (4)	C8—N1	1.290 (5)
C1—C2	1.424 (4)	C8—H8	0.9500
C2—O2	1.366 (4)	C9—N1	1.519 (6)
C2—C3	1.381 (4)	C9—H9A	0.9601
C3—C4	1.394 (5)	C9—H9B	0.9600
C3—H3	0.9500	C9'—H9C	0.9599
C4—C5	1.359 (6)	C9'—H9D	0.9600
C4—H4	0.9500	O1W—H1W	0.848 (10)
C5—C6	1.411 (5)		
O1^{i}—Co1—O1	91.99 (12)	C3—C4—H4	120.2
O1^{i}—Co1—N1^{i}	89.55 (10)	C4—C5—C6	121.8 (3)

O1—Co1—N1^{i}	153.09 (15)	C4—C5—H5	119.1
O1^{i}—Co1—N1	153.09 (15)	C6—C5—H5	119.1
O1—Co1—N1	89.55 (10)	C5—C6—C1	119.9 (3)
N1^{i}—Co1—N1	77.4 (2)	C5—C6—C8	117.1 (3)
O1^{i}—Co1—O1W	106.80 (8)	C1—C6—C8	123.1 (3)
O1—Co1—O1W	106.80 (8)	N1—C8—C6	125.9 (3)
N1^{i}—Co1—O1W	98.41 (13)	N1—C8—H8	117.0
N1—Co1—O1W	98.41 (13)	C6—C8—H8	117.0
O1—C1—C6	125.3 (3)	N1—C9—H9A	109.3
O1—C1—C2	117.8 (3)	N1—C9—H9B	109.8
C6—C1—C2	116.9 (3)	H9A—C9—H9B	108.1
O2—C2—C3	124.3 (3)	H9C—C9'—H9D	110.5
O2—C2—C1	114.1 (3)	C8—N1—C9	119.9 (4)
C3—C2—C1	121.7 (3)	C8—N1—Co1	127.0 (2)
C2—C3—C4	120.2 (3)	C9—N1—Co1	110.2 (3)
C2—C3—H3	119.9	C1—O1—Co1	128.85 (18)
C4—C3—H3	119.9	C2—O2—C7	117.2 (3)
C5—C4—C3	119.5 (3)	Co1—O1W—H1W	119.5 (17)
C5—C4—H4	120.2		

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

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O1W—H1W···O1^{ii}	0.848 (10)	2.100 (18)	2.830 (3)	144.0 (17)
O1W—H1W···O2^{ii}	0.848 (10)	2.242 (10)	2.962 (2)	142.7 (19)

Symmetry codes: (ii) *x*−1/2, −*y*+1/2, −*z*+5/2.

Fig. 1

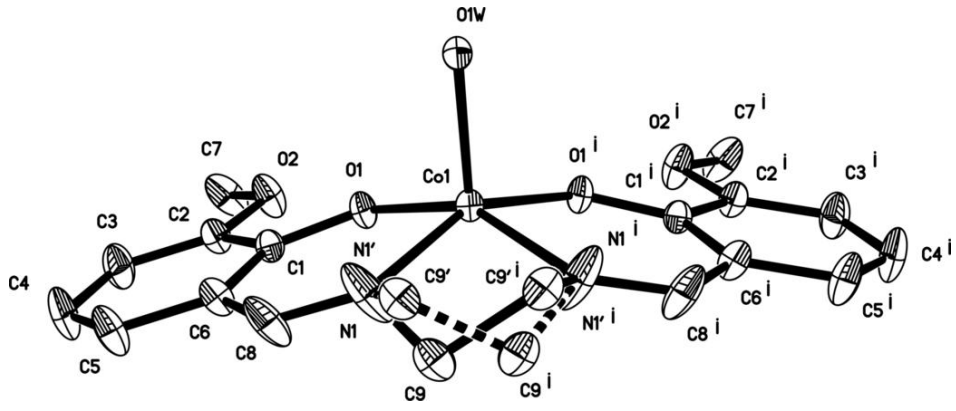
O1W
O2 i
C7 i
C7
O2
O1
Co1
O1 i
C1 i
C2 i
C3 i
C3
C2
C1
N1′
N1 i
C4 i
C4
C9′
C9′ i
C6 i
C5 i
C6
C8
N1
N1′ i
C8 i
C5
C9
C9 i

Fig. 2