Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Azido{2-[bis(2-hydroxyethyl)amino]-ethanolato-κ^4N,O,O',O''}cobalt(II)

Yan-Ju Liu,* Huai-Xia Yang, Juan Yuan and Xia Wang

Pharmacy College, Henan University of Traditional Chinese Medicine, Zhengzhou 450008, People's Republic of China
Correspondence e-mail: liuyanju886@163.com

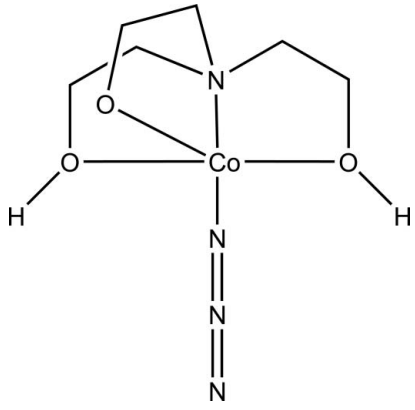
Received 8 November 2010; accepted 14 November 2010

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.038; wR factor = 0.089; data-to-parameter ratio = 16.1.

In the title complex, $[Co(C_6H_{14}NO_3)(N_3)]$ or $[Co(teaH_2)N_3]$, the Co^{II} atom resides in a trigonal–bipymidal O_3N_2 environment formed by three O atoms and one N atom from a simply deprotonated tetradentate triethanolamine ligand, and one N atom from an azide ligand. The O atoms define the equatorial plane whereas both N atoms are in axial positions. The mononuclear units are linked through O—H···O hydrogen-bonding interactions between the ethanol OH groups and the ethanolate O atom of a neighbouring complex into chains running parallel to [010].

Related literature

For general background to complexes including $teaH_3$ ligands, see: Liu, Wang *et al.* (2008); Liu, Zhang *et al.* (2008). For Co^{II} complexes with similar ligands, see: Malaestean *et al.* (2010).



Experimental

Crystal data

$[Co(C_6H_{14}NO_3)(N_3)]$
M_r = 249.14
Monoclinic, $P2_1/c$
a = 8.7752 (2) Å
b = 7.9373 (1) Å
c = 14.4097 (3) Å
β = 107.084 (1)°
V = 959.37 (3) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.78 mm^{-1}
T = 293 K
0.20 × 0.20 × 0.10 mm

Data collection

Rigaku Saturn CCD diffractometer
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)
T_{min} = 0.708, T_{max} = 0.823
4004 measured reflections
2179 independent reflections
1253 reflections with $I > 2\sigma(I)$
R_{int} = 0.055

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.089
S = 0.89
2179 reflections
135 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.38 e Å^{-3}

Table 1
Selected bond lengths (Å).

Co1—O3	1.991 (2)	Co1—O2	2.065 (2)
Co1—N2	2.013 (3)	Co1—N1	2.148 (3)
Co1—O1	2.064 (2)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O2—H1OA···O3^{i}	0.80 (6)	1.80 (6)	2.595 (3)	176.90
O1—H2OA···O3^{ii}	0.74 (3)	1.83 (3)	2.573 (3)	177.70

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

Data collection: *CrystalClear* (Rigaku/MSC, 2006); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; 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: *publCIF* (Westrip, 2010).

This study was supported by the Doctoral Research Fund of Henan Chinese Medicine (BSJJ2009–38).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: WM2427).

References

Jacobson, R. (1998). *REQAB*. Private communication to the Rigaku Corporation, Tokyo, Japan.
Liu, T., Wang, B.-W., Chen, Y.-H., Wang, Z.-M. & Gao, S. (2008). *Z. Anorg. Allg. Chem.* **634**, 778–783.
Liu, T., Zhang, Y.-J., Wang, Z.-M. & Gao, S. (2008). *J. Am. Chem. Soc.* **130**, 10500–10501.
Malaestean, I. L., Speldrich, M., Ellern, A., Baca, S. G. & Kögerler, P. (2010). *Polyhedron*, **29**, 1990–1997.
Rigaku/MSC (2006). *CrystalClear*. Rigaku/MSC, The Woodlands, Texas, USA.
Sheldrick, G. M. (2008). *Acta Cryst.* A**64**, 112–122.
Westrip, S. P. (2010). *J. Appl. Cryst.* **43**, 920–925.

supplementary materials

Acta Cryst. (2010). E**66**, m1617 [**doi:10.1107/S1600536810047100**]

Azido{2-[bis(2-hydroxyethyl)amino]ethanolato-κ^4N,O,O',O''}cobalt(II)

Y.-J. Liu, H.-X. Yang, J. Yuan and X. Wang

Comment

The design and synthesis of mononuclear compounds with strong anisotropy, potentially acting as single ion magnets, are of current interest. Podand-like or multi-dentate ligands, such as diethanolamine ($deaH_2$) or triethanolamine ($teaH_3$), have been employed though these ligands were also used to prepare other kinds of clusters (Liu, Wang *et al.*, 2008; Liu, Zhang *et al.*, 2008). In this work, we selected $teaH_3$ as a capping ligand, and azide as another anion, generating complex (I), $Co(N(CH_2CH_2OH)_2(CH_2CH_2O))N_3$ [= $Co(teaH_2)N_3$].

In the structure of (I) each Co^{II} atom is five-coordinate by three O atoms and one N atom from a simply deprotonated tetradentate triethanolamine ligand, and one N atom from an azide ligand in a trigonal-bipymidal coordination environment (Fig. 1). The O atoms define the equatorial plane whereas both N atoms sit in axial positions. The Co—N distances are 2.013 (3)—2.148 (3) Å, and the Co—O distances are 1.991 (2)–2.065 (2) Å. These bond length are in agreement with similar complexes with Co^{II} in trigonal-pyramidal coordination (Malaestean *et al.*, 2010).

The mononuclear $Co(teaH_2)N_3$ units are linked through O—H···O hydrogen bonding interactions between the ethanol OH groups and the ethanolate O atom of a neighbouring complex into chains running parallel to [010] (Fig. 2).

Experimental

Under stirring, 2.0 mmol $teaH_3$, 4.0 mmol Et_3N and 4.0 mmol NaN_3 were added, one after another, into a 20 ml methanol solution containing 1.0 mol $Co(ClO_4)_2{\cdot}6H_2O$. The resulting solution was kept stirred for another hour, and then filtered. The filtrate was allowed to stand undisturbed in a sealed vessel. Crystallization took place during one week and gave crystals in a yield of 40% based on $Co(ClO_4)_2{\cdot}6H_2O$. The product was washed with methanol and dried in air.

Refinement

H1OA and H2OA were found in difference Fourier maps and were refined freely. All other H atoms were positioned geometrically as riding atoms, with C—H = 0.97 Å and with $U_{iso}(H) = 1.2\ U_{eq}(C)$.

Figures

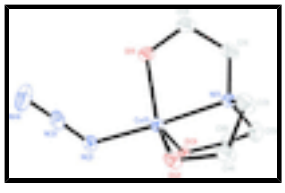


Fig. 1. View of the molecular structure of (I), showing the labelling of the atoms drawn with displacement ellipsoids at the 30% probability level. All H atoms have been omitted for clarity.

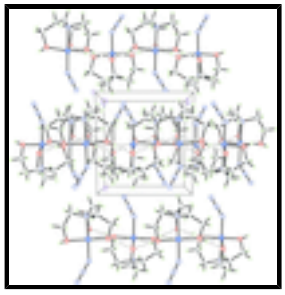


Fig. 2. A view of the crystal packing along the *c* axis. Hydrogen bonds are indicated with dashed lines.

Azido{2-[bis(2-hydroxyethyl)amino]ethanolato- κ^4*N*,*O*,*O*',*O*''}cobalt(II)

Crystal data

$[Co(C_6H_{14}NO_3)(N_3)]$	$F(000) = 516$
$M_r = 249.14$	$D_x = 1.725$ Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -P 2ybc	Cell parameters from 2179 reflections
$a = 8.7752$ (2) Å	$\theta = 3.4$–$27.5°$
$b = 7.9373$ (1) Å	$\mu = 1.78$ mm^{-1}
$c = 14.4097$ (3) Å	$T = 293$ K
$\beta = 107.084$ (1)°	Pillar, red
$V = 959.37$ (3) Å^3	0.20 × 0.20 × 0.10 mm
$Z = 4$	

Data collection

Rigaku Saturn CCD diffractometer	2179 independent reflections
Radiation source: fine-focus sealed tube	1253 reflections with $I > 2\sigma(I)$
graphite	$R_{int} = 0.055$
Detector resolution: 0.76 pixels mm^{-1}	$\theta_{max} = 27.5°$, $\theta_{min} = 3.5°$
ω scans	$h = -11 \rightarrow 11$
Absorption correction: multi-scan (*REQAB*; Jacobson, 1998)	$k = -10 \rightarrow 10$
$T_{min} = 0.708$, $T_{max} = 0.823$	$l = -18 \rightarrow 18$
4004 measured reflections	

Refinement

Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)] = 0.038$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.089$	H atoms treated by a mixture of independent and constrained refinement
$S = 0.89$	$w = 1/[\sigma^2(F_o^2) + (0.0427P)^2]$

	where $P = (F_o^2 + 2F_c^2)/3$
2179 reflections	$(\Delta/\sigma)_{max} = 0.001$
135 parameters	$\Delta\rho_{max} = 0.55$ e Å^{-3}
0 restraints	$\Delta\rho_{min} = -0.38$ e Å^{-3}

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}
Co1	0.49520 (5)	0.87496 (5)	0.81512 (3)	0.02597 (16)
C1	0.2117 (4)	0.6897 (4)	0.8302 (3)	0.0438 (10)
H1A	0.1899	0.6223	0.7716	0.053*
H1B	0.1173	0.6886	0.8522	0.053*
C2	0.3480 (4)	0.6158 (5)	0.9063 (3)	0.0405 (9)
H2A	0.3523	0.6638	0.9690	0.049*
H2B	0.3329	0.4951	0.9096	0.049*
C3	0.2225 (5)	0.9829 (4)	0.8818 (3)	0.0424 (10)
H3A	0.2555	0.9297	0.9451	0.051*
H3B	0.1099	1.0092	0.8665	0.051*
C4	0.3151 (4)	1.1428 (4)	0.8848 (3)	0.0362 (9)
H4A	0.2619	1.2139	0.8301	0.043*
H4B	0.3217	1.2040	0.9441	0.043*
C5	0.1604 (4)	0.9162 (5)	0.7086 (3)	0.0423 (10)
H5A	0.1562	1.0382	0.7049	0.051*
H5B	0.0518	0.8745	0.6925	0.051*
C6	0.2386 (4)	0.8496 (5)	0.6363 (2)	0.0361 (9)
H6A	0.2138	0.7309	0.6249	0.043*
H6B	0.1970	0.9086	0.5751	0.043*
N1	0.2476 (3)	0.8648 (3)	0.80846 (19)	0.0254 (6)
N2	0.7314 (3)	0.8811 (4)	0.8330 (2)	0.0405 (7)
N3	0.8273 (4)	0.8233 (4)	0.9028 (2)	0.0388 (8)
N4	0.9226 (5)	0.7663 (5)	0.9675 (3)	0.0703 (12)
O1	0.4947 (3)	0.6485 (3)	0.88538 (18)	0.0332 (6)
O2	0.4718 (3)	1.1008 (3)	0.88087 (18)	0.0344 (6)
O3	0.4087 (2)	0.8714 (3)	0.67098 (14)	0.0269 (5)
H1OA	0.508 (6)	1.183 (7)	0.863 (4)	0.11 (2)*
H2OA	0.525 (4)	0.569 (4)	0.870 (3)	0.035 (12)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0262 (2)	0.0243 (2)	0.0272 (2)	−0.0003 (2)	0.00739 (18)	0.0007 (2)
C1	0.040 (2)	0.0305 (19)	0.064 (3)	−0.0069 (17)	0.021 (2)	0.0004 (18)
C2	0.050 (2)	0.0339 (19)	0.046 (2)	0.002 (2)	0.0274 (19)	0.0055 (19)
C3	0.044 (2)	0.034 (2)	0.059 (2)	−0.0046 (18)	0.030 (2)	−0.0111 (19)
C4	0.042 (2)	0.0266 (19)	0.048 (2)	−0.0025 (18)	0.0257 (18)	−0.0068 (17)
C5	0.029 (2)	0.054 (3)	0.043 (2)	0.0035 (18)	0.0095 (17)	−0.0026 (18)
C6	0.0263 (19)	0.045 (2)	0.0335 (19)	−0.0002 (17)	0.0027 (16)	−0.0022 (17)
N1	0.0282 (14)	0.0220 (13)	0.0280 (14)	−0.0013 (13)	0.0114 (12)	−0.0021 (12)
N2	0.0264 (16)	0.0488 (18)	0.0465 (19)	0.0014 (16)	0.0112 (15)	0.0126 (17)
N3	0.0272 (18)	0.047 (2)	0.045 (2)	−0.0005 (15)	0.0145 (16)	−0.0082 (16)
N4	0.046 (2)	0.107 (3)	0.050 (2)	0.024 (2)	0.001 (2)	0.008 (2)
O1	0.0377 (15)	0.0227 (15)	0.0420 (15)	0.0045 (12)	0.0163 (12)	0.0026 (12)
O2	0.0372 (15)	0.0274 (14)	0.0411 (14)	−0.0091 (12)	0.0152 (11)	−0.0053 (12)
O3	0.0265 (12)	0.0302 (12)	0.0252 (11)	0.0039 (12)	0.0093 (9)	0.0022 (11)

Geometric parameters (Å, °)

Co1—O3	1.991 (2)	C3—H3B	0.9700
Co1—N2	2.013 (3)	C4—O2	1.433 (4)
Co1—O1	2.064 (2)	C4—H4A	0.9700
Co1—O2	2.065 (2)	C4—H4B	0.9700
Co1—N1	2.148 (3)	C5—N1	1.475 (4)
C1—N1	1.478 (4)	C5—C6	1.502 (5)
C1—C2	1.486 (5)	C5—H5A	0.9700
C1—H1A	0.9700	C5—H5B	0.9700
C1—H1B	0.9700	C6—O3	1.439 (4)
C2—O1	1.430 (4)	C6—H6A	0.9700
C2—H2A	0.9700	C6—H6B	0.9700
C2—H2B	0.9700	N2—N3	1.197 (4)
C3—N1	1.476 (4)	N3—N4	1.147 (4)
C3—C4	1.501 (5)	O1—H2OA	0.74 (3)
C3—H3A	0.9700	O2—H1OA	0.81 (5)
O3—Co1—N2	101.32 (11)	O2—C4—H4B	110.0
O3—Co1—O1	116.37 (10)	C3—C4—H4B	110.0
N2—Co1—O1	96.24 (12)	H4A—C4—H4B	108.3
O3—Co1—O2	115.60 (10)	N1—C5—C6	111.6 (3)
N2—Co1—O2	99.08 (12)	N1—C5—H5A	109.3
O1—Co1—O2	121.07 (10)	C6—C5—H5A	109.3
O3—Co1—N1	83.24 (9)	N1—C5—H5B	109.3
N2—Co1—N1	175.37 (11)	C6—C5—H5B	109.3
O1—Co1—N1	80.87 (10)	H5A—C5—H5B	108.0
O2—Co1—N1	79.50 (10)	O3—C6—C5	110.8 (3)
N1—C1—C2	110.6 (3)	O3—C6—H6A	109.5
N1—C1—H1A	109.5	C5—C6—H6A	109.5

C2—C1—H1A	109.5	O3—C6—H6B	109.5
N1—C1—H1B	109.5	C5—C6—H6B	109.5
C2—C1—H1B	109.5	H6A—C6—H6B	108.1
H1A—C1—H1B	108.1	C5—N1—C3	112.2 (3)
O1—C2—C1	110.6 (3)	C5—N1—C1	112.6 (3)
O1—C2—H2A	109.5	C3—N1—C1	111.0 (3)
C1—C2—H2A	109.5	C5—N1—Co1	105.15 (19)
O1—C2—H2B	109.5	C3—N1—Co1	107.8 (2)
C1—C2—H2B	109.5	C1—N1—Co1	107.6 (2)
H2A—C2—H2B	108.1	N3—N2—Co1	122.8 (2)
N1—C3—C4	111.4 (3)	N4—N3—N2	177.5 (4)
N1—C3—H3A	109.4	C2—O1—Co1	113.2 (2)
C4—C3—H3A	109.4	C2—O1—H2OA	109 (3)
N1—C3—H3B	109.4	Co1—O1—H2OA	123 (3)
C4—C3—H3B	109.4	C4—O2—Co1	116.5 (2)
H3A—C3—H3B	108.0	C4—O2—H1OA	107 (4)
O2—C4—C3	108.7 (3)	Co1—O2—H1OA	117 (4)
O2—C4—H4A	110.0	C6—O3—Co1	113.77 (18)
C3—C4—H4A	110.0		

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H1OA···O3^{i}	0.80 (6)	1.80 (6)	2.595 (3)	176.90
O1—H2OA···O3^{ii}	0.74 (3)	1.83 (3)	2.573 (3)	177.70

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

Fig. 1

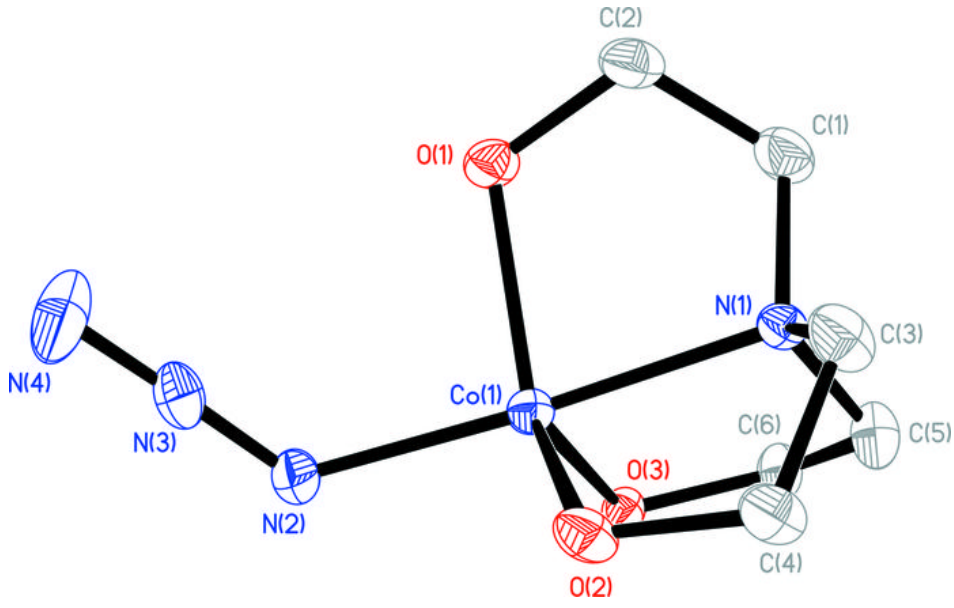


Fig. 2