Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

Bis[2-(ethylamino)ethanol-κ^2N,O]-bis(saccharinato-κN)cadmium(II)

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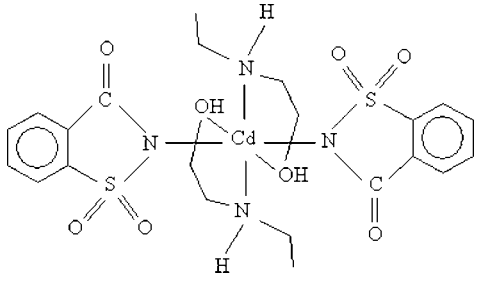
Received 5 November 2007; accepted 5 November 2007

Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.018 Å; R factor = 0.049; wR factor = 0.135; data-to-parameter ratio = 17.8.

In the centrosymmetric title complex, $[Cd(C_7H_4NO_3S)_2(C_4H_{11}NO)_2]$, the Cd^{II} ion is coordinated by two saccharinate (sac) anions and two N,O-bidentate 2-(ethylamino)ethanol ligands, displaying a distorted octahedral coordination. Pairs of molecules are doubly bridged by O—H···O hydrogen bonds and these dimers are further linked by weak C—H···π(sac) interactions.

Related literature

For related structures, see: Ahlgrén *et al.* (1982); Turpeinen & Hamalainen (1985); Turpeinen *et al.* (1996); Vinogradova *et al.* (2001); Yilmaz *et al.* (2006); Baran & Yilmaz (2006); Yilmaz *et al.* (2007).



Experimental

Crystal data

$[Cd(C_7H_4NO_3S)_2(C_4H_{11}NO)_2]$
M_r = 655.05
Monoclinic, $P2_1/c$
a = 7.7961 (6) Å
b = 19.2676 (19) Å
c = 8.8162 (7) Å
β = 93.061 (6)°
V = 1322.4 (2) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.04 mm^{-1}
T = 296 (2) K
0.21 × 0.19 × 0.18 mm

Data collection

STOE IPDS 2 diffractometer
Absorption correction: integration (*X-RED32*; Stoe & Cie, 2002)
T_{min} = 0.783, T_{max} = 0.822
21045 measured reflections
2980 independent reflections
2013 reflections with $I > 2\sigma(I)$
R_{int} = 0.076

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.049
$wR(F^2)$ = 0.135
S = 0.96
2980 reflections
167 parameters
3 restraints
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 1.42 e Å^{-3}
$\Delta\rho_{min}$ = −1.17 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cd1—N1	2.397 (9)	Cd1—O4	2.317 (8)
Cd1—N2	2.267 (10)		

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

D—H···A	D—H	H···A	D···A	D—H···A
O4—H4A···O3^{i}	0.89 (10)	1.99 (6)	2.863 (11)	168 (15)
N2—H2···O1	0.91	2.49	3.336 (16)	154

Symmetry code: (i) $-x-1, -y, -z$.

Data collection: *X-AREA* (Stoe & Cie, 2002); cell refinement: *X-AREA*; data reduction: *X-RED32* (Stoe & Cie, 2002); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2635).

References

Ahlgrén, M., Turpeinen, U. & Hämäläinen, R. (1982). *Acta Cryst.* B**38**, 429–433.
Baran, E. J. & Yilmaz, V. T. (2006). *Coord. Chem. Rev.* **250**, 1980–1999.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Stoe & Cie (2002). *X-AREA* (Version 1.18) and *X-RED32* (Version 1.04). Stoe & Cie. Darmstadt, Germany.
Turpeinen, U. Hämäläinen, R. & Ahlgrén, M. (1985). *Acta Cryst.* C**41**, 1728–1730.
Turpeinen, U., Hämäläinen, R., Mutikainen, I. & Orama, O. (1996). *Acta Cryst.* C**52**, 568–570.
Vinogradova, E. A., Vassilyeva, O. Y., Kokozay, V. N. & Skelton, V. (2001). *Z. Naturforsch. Teil B*, **57**, 319–322.
Yilmaz, V. T., Kars, V. & Kazak, C. (2006). *Z. Naturforsch. Teil B*, **61**, 555–559.
Yilmaz, V. T., Kars, V. & Kazak, C. (2007). *Z. Anorg. Allg. Chem.* **633**, 351–353.

 doi:10.1107/S1600536807056036

supplementary materials

Acta Cryst. (2007). E**63**, m2947 [**doi:10.1107/S1600536807056036**]

Bis[2-(ethylamino)ethanol-κ^2N,O]bis(saccharinato-κN)cadmium(II)

V. T. Yilmaz and C. Kazak

Comment

Although metal complexes of 2-dimethylaminoethanol have received considerable attention (Ahlgrén *et al.* 1982; Turpeinen & Hamalainen 1985; Turpeinen *et al.*, 1996; Vinogradova *et al.*, 2001), 2-ethylaminoethanol-metal complexes are very rare and we recently reported the first two copper(II) complexes (Yilmaz *et al.*, 2006, 2007). This work is a part of our study on the synthesis and structural characterization of metal complexes of saccharin (Hsac) with other co-ligands (Baran & Yilmaz 2006). We report here the crystal and molecular structure of the title compound, (I).

As illustrated in (Fig. 1), (I) is a mononuclear Cd^{II} complex, in which the Cd^{II} ion lies on a centre of inversion and exhibits a somewhat distorted octahedral coordination geometry with two neutral bidendate (N, O) eae ligands and two anionic sac ligands (Table 1). The amine hydrogen atoms of 2-ethyaminoethanol form intramolecular N—H···O interactions with sulfonyl O atoms of the saccharinate anion (Table 2). Individual molecules are linked into pairs by double O—H···O hydrogen bonds involving the H atoms of the hydroxyl groups of 2-ethyaminoethanol and the carbonyl O atoms of the saccharinate anion (Fig. 2). The dimeric units are further linked by weak C—H···π-(sac) interactions with an H···π separation of 2.97 Å.

Experimental

2-Ethylaminoethanol (0.09 g, 1 mmol) was added dropwise to a 20-ml methanol solution containing $Cd(OAc)_2{\cdot}2H_2O$ (0.13 g, 0.5 mmol) and saccharin (0.18 g, 1 mmol). The reaction solution was stirred for 30 min at room temperature. Colourless prisms of (I) were obtained after 2 days by slow evoparation of the solution at room temperature.

Refinement

The O-bound H atom was located in a difference map and freely refined. The other H atoms were refined with a riding model (C—H = 0.93–0.97 Å, N—H = 0.91 Å) with U_{iso}(H) = 1.2 U_{eq}(C, N) or 1.5U_{eq}(methyl C).

Figures

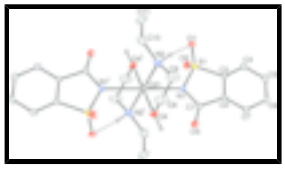


Fig. 1. The molecular structure of (I) showing 50% displacement ellipsoids (arbitrary spheres for the H atoms). Symmetry code: (i) −*x*, −*y*, −*z*. The intermolecular N—H···O bonds are indicated by dashed lines.

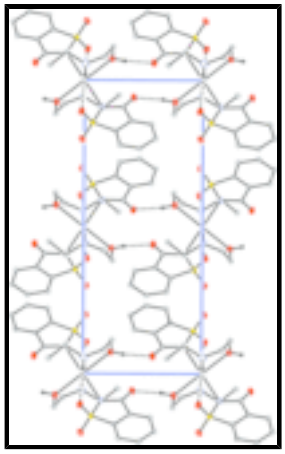


Fig. 2. The packing of the molecules in (I) along the *b* axis. Dashed lines represent hydrogen bonds.

Bis[2-(ethylamino)ethanol-κ^2*N*,*O*]bis(saccharinato-κ*N*)cadmium(II)

Crystal data

$[Cd(C_7H_4NO_3S)_2(C_4H_{11}NO)_2]$	F_{000} = 668
M_r = 655.05	D_x = 1.645 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71069 Å
Hall symbol: -P 2ybc	Cell parameters from 21565 reflections
a = 7.7961 (6) Å	θ = 2.1–27.4º
b = 19.2676 (19) Å	μ = 1.04 mm^{-1}
c = 8.8162 (7) Å	T = 296 (2) K
β = 93.061 (6)º	Prism, colorless
V = 1322.4 (2) Å^3	0.21 × 0.19 × 0.18 mm
Z = 2	

Data collection

STOE IPDS 2 diffractometer	2980 independent reflections
Radiation source: fine-focus sealed tube	2013 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.076
Detector resolution: 6.67 pixels mm^{-1}	θ_{max} = 27.4º
T = 296(2) K	θ_{min} = 2.1º
rotation method scans	h = −10→9
Absorption correction: integration (X-RED32; Stoe & Cie, 2002)	k = −24→24
T_{min} = 0.783, T_{max} = 0.822	l = −11→11
21045 measured reflections	

Refinement

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

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.049$

$wR(F^2) = 0.135$

$S = 0.96$

2980 reflections

167 parameters

3 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

$w = 1/[\sigma^2(F_o^2) + (0.085P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

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

$\Delta\rho_{max} = 1.42$ e Å^{-3}

$\Delta\rho_{min} = -1.17$ 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}
C1	−0.3311 (13)	0.0910 (6)	−0.1374 (13)	0.038 (2)
C2	−0.3971 (12)	0.1381 (5)	−0.2668 (11)	0.032 (2)
C3	−0.2683 (13)	0.1732 (5)	−0.3368 (11)	0.034 (2)
C4	−0.3022 (16)	0.2207 (6)	−0.4505 (13)	0.047 (3)
H4	−0.2144	0.2443	−0.4959	0.056*
C5	−0.4724 (16)	0.2318 (7)	−0.4944 (15)	0.056 (3)
H5	−0.4997	0.2634	−0.5717	0.067*
C6	−0.6030 (16)	0.1975 (6)	−0.4271 (14)	0.048 (3)
H6	−0.7164	0.2063	−0.4593	0.058*
C7	−0.5676 (13)	0.1499 (6)	−0.3118 (12)	0.039 (2)
H7	−0.6556	0.1265	−0.2661	0.047*
C8	−0.242 (3)	−0.1147 (11)	−0.139 (2)	0.096 (5)
H8A	−0.1807	−0.1577	−0.1192	0.115*
H8B	−0.3614	−0.1261	−0.1656	0.115*
C9	−0.174 (3)	−0.0818 (12)	−0.261 (2)	0.096 (5)
H9A	−0.2485	−0.0438	−0.2930	0.115*
H9B	−0.1698	−0.1143	−0.3444	0.115*
C10	0.152 (2)	−0.0957 (8)	−0.2728 (17)	0.073 (4)
H10A	0.1965	−0.1225	−0.1863	0.088*
H10B	0.1171	−0.1282	−0.3527	0.088*

C11	0.290 (2)	−0.0513 (11)	−0.327 (2)	0.092 (6)
H11A	0.3867	−0.0796	−0.3498	0.137*
H11B	0.3247	−0.0185	−0.2488	0.137*
H11C	0.2494	−0.0268	−0.4163	0.137*
N1	−0.1521 (11)	0.0927 (5)	−0.1290 (10)	0.039 (2)
N2	−0.0067 (15)	−0.0556 (7)	−0.2268 (12)	0.063 (3)
H2	−0.0052	−0.0189	−0.2918	0.075*
O1	0.0239 (10)	0.1062 (5)	−0.3575 (10)	0.051 (2)
O2	0.0178 (10)	0.2013 (4)	−0.1787 (11)	0.054 (2)
O3	−0.4149 (10)	0.0592 (5)	−0.0628 (11)	0.056 (2)
O4	−0.2338 (10)	−0.0744 (5)	−0.0105 (10)	0.051 (2)
S1	−0.0711 (3)	0.14465 (14)	−0.2516 (3)	0.0373 (6)
Cd1	0.0000	0.0000	0.0000	0.0364 (4)
H4A	−0.337 (10)	−0.066 (8)	0.024 (17)	0.07 (5)*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.031 (5)	0.040 (5)	0.044 (6)	−0.001 (4)	0.003 (5)	0.004 (5)
C2	0.032 (5)	0.031 (5)	0.032 (5)	0.000 (4)	0.001 (4)	0.000 (4)
C3	0.036 (5)	0.034 (5)	0.031 (5)	0.002 (4)	0.004 (4)	0.002 (4)
C4	0.054 (7)	0.044 (6)	0.042 (6)	0.000 (5)	0.008 (5)	0.014 (5)
C5	0.061 (9)	0.055 (7)	0.050 (7)	0.008 (6)	−0.006 (6)	0.022 (6)
C6	0.040 (6)	0.053 (7)	0.050 (7)	0.005 (5)	−0.009 (5)	0.004 (5)
C7	0.033 (5)	0.042 (6)	0.043 (6)	−0.005 (4)	0.002 (4)	0.000 (5)
C8	0.089 (9)	0.120 (11)	0.082 (9)	−0.050 (8)	0.021 (7)	−0.040 (8)
C9	0.089 (9)	0.120 (11)	0.082 (9)	−0.050 (8)	0.021 (7)	−0.040 (8)
C10	0.110 (13)	0.061 (9)	0.048 (8)	0.016 (9)	0.005 (8)	−0.011 (6)
C11	0.052 (9)	0.131 (17)	0.093 (13)	0.014 (10)	0.009 (9)	−0.036 (12)
N1	0.027 (4)	0.046 (5)	0.043 (5)	0.002 (4)	0.003 (4)	0.016 (4)
N2	0.062 (7)	0.088 (8)	0.039 (6)	−0.018 (6)	0.003 (5)	−0.014 (5)
O1	0.040 (4)	0.061 (5)	0.052 (5)	0.011 (4)	0.018 (4)	0.007 (4)
O2	0.042 (4)	0.052 (5)	0.067 (6)	−0.011 (4)	0.000 (4)	0.003 (4)
O3	0.035 (4)	0.067 (5)	0.068 (6)	−0.001 (4)	0.012 (4)	0.035 (5)
O4	0.035 (4)	0.057 (5)	0.061 (5)	−0.001 (4)	0.006 (4)	0.003 (4)
S1	0.0281 (12)	0.0427 (13)	0.0417 (14)	0.0019 (11)	0.0068 (10)	0.0069 (11)
Cd1	0.0379 (6)	0.0371 (6)	0.0339 (6)	−0.0007 (5)	−0.0005 (4)	0.0030 (5)

Geometric parameters (Å, °)

C1—O3	1.133 (12)	C9—H9B	0.9700
C1—N1	1.394 (13)	C10—C11	1.48 (3)
C1—C2	1.525 (14)	C10—N2	1.53 (2)
C2—C3	1.383 (14)	C10—H10A	0.9700
C2—C7	1.386 (14)	C10—H10B	0.9700
C3—C4	1.372 (14)	C11—H11A	0.9600
C3—S1	1.763 (10)	C11—H11B	0.9600
C4—C5	1.379 (17)	C11—H11C	0.9600
C4—H4	0.9300	N1—S1	1.625 (9)

C5—C6	1.375 (18)	N2—H2	0.9100
C5—H5	0.9300	O1—S1	1.429 (8)
C6—C7	1.386 (16)	O2—S1	1.427 (9)
C6—H6	0.9300	O4—H4A	0.89 (10)
C7—H7	0.9300	Cd1—N2^{i}	2.267 (10)
C8—C9	1.37 (3)	Cd1—O4^{i}	2.317 (8)
C8—O4	1.374 (18)	Cd1—N1^{i}	2.397 (9)
C8—H8A	0.9700	Cd1—N1	2.397 (9)
C8—H8B	0.9700	Cd1—N2	2.267 (10)
C9—N2	1.41 (2)	Cd1—O4	2.317 (8)
C9—H9A	0.9700		
O3—C1—N1	126.0 (11)	C10—C11—H11A	109.5
O3—C1—C2	125.1 (10)	C10—C11—H11B	109.5
N1—C1—C2	108.8 (8)	H11A—C11—H11B	109.5
C3—C2—C7	120.0 (9)	C10—C11—H11C	109.5
C3—C2—C1	113.6 (9)	H11A—C11—H11C	109.5
C7—C2—C1	126.3 (9)	H11B—C11—H11C	109.5
C4—C3—C2	122.4 (10)	C1—N1—S1	113.7 (7)
C4—C3—S1	130.5 (8)	C1—N1—Cd1	118.4 (7)
C2—C3—S1	107.1 (7)	S1—N1—Cd1	125.1 (5)
C3—C4—C5	117.0 (11)	C9—N2—C10	120.7 (13)
C3—C4—H4	121.5	C9—N2—Cd1	109.4 (9)
C5—C4—H4	121.5	C10—N2—Cd1	119.5 (9)
C6—C5—C4	121.8 (11)	C9—N2—H2	100.8
C6—C5—H5	119.1	C10—N2—H2	100.8
C4—C5—H5	119.1	Cd1—N2—H2	100.8
C5—C6—C7	120.8 (11)	C8—O4—Cd1	112.7 (8)
C5—C6—H6	119.6	C8—O4—H4A	112 (10)
C7—C6—H6	119.6	Cd1—O4—H4A	127 (10)
C2—C7—C6	117.9 (10)	O2—S1—O1	115.8 (5)
C2—C7—H7	121.0	O2—S1—N1	111.6 (5)
C6—C7—H7	121.0	O1—S1—N1	110.4 (5)
C9—C8—O4	112.3 (15)	O2—S1—C3	110.2 (5)
C9—C8—H8A	109.1	O1—S1—C3	110.7 (5)
O4—C8—H8A	109.1	N1—S1—C3	96.6 (5)
C9—C8—H8B	109.1	N2^{i}—Cd1—N2	180.0 (9)
O4—C8—H8B	109.1	N2^{i}—Cd1—O4^{i}	72.0 (4)
H8A—C8—H8B	107.9	N2—Cd1—O4^{i}	108.0 (4)
C8—C9—N2	113.2 (17)	N2^{i}—Cd1—O4	108.0 (4)
C8—C9—H9A	108.9	N2—Cd1—O4	72.0 (4)
N2—C9—H9A	108.9	O4^{i}—Cd1—O4	180.0 (4)
C8—C9—H9B	108.9	N2^{i}—Cd1—N1^{i}	86.9 (4)
N2—C9—H9B	108.9	N2—Cd1—N1^{i}	93.1 (4)
H9A—C9—H9B	107.8	O4^{i}—Cd1—N1^{i}	94.3 (3)
C11—C10—N2	114.0 (12)	O4—Cd1—N1^{i}	85.7 (3)
C11—C10—H10A	108.8	N2^{i}—Cd1—N1	93.1 (4)

N2—C10—H10A	108.8	N2—Cd1—N1	86.9 (4)
C11—C10—H10B	108.8	O4^{i}—Cd1—N1	85.7 (3)
N2—C10—H10B	108.8	O4—Cd1—N1	94.3 (3)
H10A—C10—H10B	107.6	N1^{i}—Cd1—N1	180.0 (5)

Symmetry codes: (i) −*x*, −*y*, −*z*.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O4—H4A···O3^{ii}	0.89 (10)	1.99 (6)	2.863 (11)	168 (15)
N2—H2···O1	0.91	2.49	3.336 (16)	154

Symmetry codes: (ii) −*x*−1, −*y*, −*z*.

Fig. 1

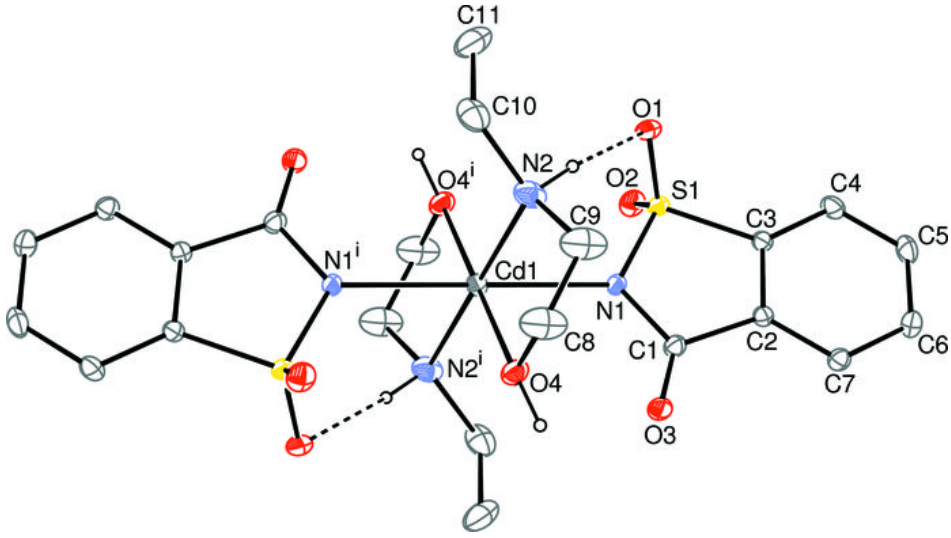

C11
C10
O1
N2
O4i
O2
S1
C4
C9
C3
C5
N1i
Cd1
N1
C8
C1
C2
C6
N2i
O4
C7
O3

Fig. 2