

Bis{2-[2-(dimethylamino)ethylimino-methyl]-4,6-disulfanylphenolato}-cobalt(II) monohydrate

Ying-Jie Cai, Jin Li, Peng Huang and Qing-Fu Zeng*

 Engineering Research Center for Clean Production of Textile Dyeing and Printing, Ministry of Education, Wuhan 430073, People's Republic of China
 Correspondence e-mail: qfzeng@wuse.edu.cn

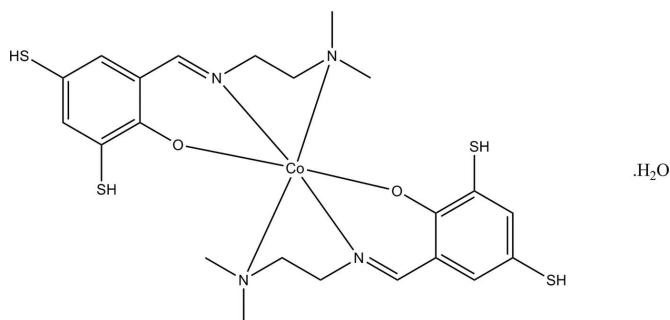
Received 20 August 2009; accepted 24 August 2009

 Key indicators: single-crystal X-ray study; $T = 296$ K; mean $\sigma(\text{C}-\text{C}) = 0.005$ Å; R factor = 0.054; wR factor = 0.186; data-to-parameter ratio = 15.9.

In the title hydrated complex, $[\text{Co}(\text{C}_{11}\text{H}_{15}\text{N}_2\text{OS}_2)_2]\cdot\text{H}_2\text{O}$, the Co^{II} atom (site symmetry 2) is coordinated by two O,N,N' -tridentate Schiff base ligands, resulting in a very distorted $cis\text{-CoO}_2\text{N}_4$ octahedral geometry for the metal ion. In the crystal, the water molecule (O-atom site symmetry 2) interacts with nearby complex molecules by way of bifurcated $\text{O}-\text{H}\cdots(\text{O},\text{S})$ hydrogen bonds.

Related literature

For a related compound and background, see: Li *et al.* (2009).
 For reference structural data, see: Allen *et al.* (1987).



Experimental

Crystal data

$[\text{Co}(\text{C}_{11}\text{H}_{15}\text{N}_2\text{OS}_2)_2]\cdot\text{H}_2\text{O}$
 $M_r = 587.69$
 Orthorhombic, $Pbcn$
 $a = 12.3755$ (15) Å
 $b = 9.2485$ (15) Å
 $c = 22.827$ (3) Å

$V = 2612.7$ (6) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.01$ mm⁻¹
 $T = 296$ K
 $0.30 \times 0.25 \times 0.25$ mm

Data collection

Enraf–Nonius CAD-4
 diffractometer
 Absorption correction: ψ scan
 (North *et al.*, 1968)
 $T_{\text{min}} = 0.752$, $T_{\text{max}} = 0.787$
 13847 measured reflections

2548 independent reflections
 2039 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.031$
 200 standard reflections
 every 3 reflections
 intensity decay: 1%

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.054$
 $wR(F^2) = 0.186$
 $S = 1.06$
 2548 reflections
 160 parameters
 1 restraint

H atoms treated by a mixture of independent and constrained refinement
 $\Delta\rho_{\text{max}} = 0.84$ e Å⁻³
 $\Delta\rho_{\text{min}} = -0.96$ e Å⁻³

Table 1

Selected bond lengths (Å).

Co1–O1	2.089 (2)	Co1–N1	2.456 (3)
Co1–N2	2.241 (3)		

Table 2

Hydrogen-bond geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$\text{O2}-\text{H2A}\cdots\text{S2}^i$	0.832 (10)	2.95 (2)	3.7001 (17)	151 (4)
$\text{O2}-\text{H2A}\cdots\text{O1}^i$	0.832 (10)	2.25 (3)	2.928 (5)	139 (4)

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

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD4* (Harms & Wocadlo, 1995); 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: *SHELXTL*.

The project was supported by the Scientific Research Foundation for Returned Overseas Chinese Scholars, State Education Ministry, Educational Commission of Hubei Province (D20091703) and the Natural Science Foundation of Hubei Province (2008CDB038).

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5056).

References

- Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
 Enraf–Nonius (1989). *CAD-4 Software*. Enraf–Nonius, Delft, The Netherlands.
 Harms, K. & Wocadlo, S. (1995). *XCAD4*. University of Marburg, Germany.
 Li, H.-Y., Wang, L.-J., Hou, J. & Zeng, Q.-F. (2009). *Acta Cryst. E* **65**, m1112.
 North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst. A* **24**, 351–359.
 Sheldrick, G. M. (2008). *Acta Cryst. A* **64**, 112–122.

supplementary materials

Acta Cryst. (2009). E65, m1146 [doi:10.1107/S1600536809033650]

Bis{2-[2-(dimethylamino)ethyliminomethyl]-4,6-disulfanylphenolato}cobalt(II) monohydrate

Y.-J. Cai, J. Li, P. Huang and Q.-F. Zeng

Comment

As part of our ongoing studies of this family of compounds (Li *et al.*, 2009), we report here the crystal structure of the title compound, (I). In (I), all bond lengths are within normal ranges (Allen *et al.*, 1987) (Fig. 1). The Co(II) is six-coordinated in a distorted octahedral coordination by two N,N,O-tridentate Schiff base ligands.

Experimental

A mixture of 2-hydroxy-3,5-dimercaptobenzaldehyde (372 mg, 2 mmol), *N,N*-dimethylethane-1,2-diamine (176 mg, 2 mmol) and $\text{CoCl}_2 \cdot 6\text{H}_2\text{O}$ (1 mmol, 238 mg) in methanol (10 ml) was stirred for 1 h. After keeping the filtrate in air for 7 d, red blocks of (I) were formed.

Refinement

The water H atom was located in a difference map and its position was freely refined. All other H atoms were positioned geometrically (C—H = 0.93–0.97 Å, S—H = 1.2 Å) and refined as riding, with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{carrier})$ or $1.5U_{\text{eq}}(\text{methyl C})$.

Figures

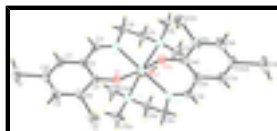


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids. Atoms with the suffix A are generated by the symmetry operation $(1-x, y, 3/2-z)$.

Bis{2-[2-(dimethylamino)ethyliminomethyl]-4,6-disulfanylphenolato}cobalt(II) monohydrate

Crystal data

$[\text{Co}(\text{C}_{11}\text{H}_{15}\text{N}_2\text{OS}_2)_2] \cdot \text{H}_2\text{O}$

$M_r = 587.69$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 12.3755$ (15) Å

$b = 9.2485$ (15) Å

$c = 22.827$ (3) Å

$V = 2612.7$ (6) Å³

$Z = 4$

$F_{000} = 1228$

$D_x = 1.494$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 25 reflections

$\theta = 9\text{--}12^\circ$

$\mu = 1.01$ mm⁻¹

$T = 296$ K

Block, red

$0.30 \times 0.25 \times 0.25$ mm

Data collection

Enraf–Nonius CAD-4 diffractometer	$R_{\text{int}} = 0.031$
Radiation source: fine-focus sealed tube	$\theta_{\text{max}} = 26.0^\circ$
Monochromator: graphite	$\theta_{\text{min}} = 2.4^\circ$
$T = 296$ K	$h = -15 \rightarrow 15$
$\omega/2\theta$ scans	$k = -11 \rightarrow 6$
Absorption correction: ψ scan (North <i>et al.</i> , 1968)	$l = -28 \rightarrow 27$
$T_{\text{min}} = 0.752$, $T_{\text{max}} = 0.787$	200 standard reflections
13847 measured reflections	every 3 reflections
2548 independent reflections	intensity decay: 1%
2039 reflections with $I > 2\sigma(I)$	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.054$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.186$	$w = 1/[\sigma^2(F_o^2) + (0.1201P)^2 + 2.1692P]$
$S = 1.06$	where $P = (F_o^2 + 2F_c^2)/3$
2548 reflections	$(\Delta/\sigma)_{\text{max}} = 0.001$
160 parameters	$\Delta\rho_{\text{max}} = 0.84 \text{ e } \text{\AA}^{-3}$
1 restraint	$\Delta\rho_{\text{min}} = -0.96 \text{ e } \text{\AA}^{-3}$
Primary atom site location: structure-invariant direct methods	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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
C3	0.2776 (3)	0.1848 (3)	0.68222 (16)	0.0417 (8)

H3	0.2082	0.2207	0.6768	0.050*
C4	0.4167 (3)	0.0107 (3)	0.64302 (14)	0.0380 (7)
C5	0.3168 (3)	0.0854 (4)	0.63802 (14)	0.0395 (7)
C6	0.4439 (3)	-0.0791 (4)	0.59437 (15)	0.0467 (9)
C7	0.2502 (3)	0.0634 (4)	0.58895 (17)	0.0524 (10)
H7	0.1846	0.1120	0.5866	0.063*
C8	0.2678 (3)	0.3286 (4)	0.76616 (19)	0.0507 (9)
H8A	0.2245	0.2739	0.7938	0.061*
H8B	0.2195	0.3881	0.7429	0.061*
C9	0.3788 (4)	-0.0974 (4)	0.54666 (16)	0.0588 (11)
H9	0.4004	-0.1560	0.5157	0.071*
C10	0.2796 (4)	-0.0272 (5)	0.54503 (18)	0.0623 (12)
C11	0.3458 (3)	0.4236 (4)	0.79892 (18)	0.0514 (9)
H11A	0.3791	0.4909	0.7718	0.062*
H11B	0.3066	0.4795	0.8279	0.062*
C14	0.5090 (4)	0.4381 (5)	0.8533 (2)	0.0673 (13)
H14A	0.5339	0.5032	0.8234	0.101*
H14B	0.5692	0.3847	0.8686	0.101*
H14C	0.4757	0.4922	0.8842	0.101*
C15	0.3836 (4)	0.2501 (5)	0.87496 (19)	0.0651 (12)
H15A	0.3375	0.3091	0.8989	0.098*
H15B	0.4402	0.2100	0.8987	0.098*
H15C	0.3421	0.1732	0.8579	0.098*
Co1	0.5000	0.18183 (7)	0.7500	0.0390 (3)
H2A	0.502 (3)	0.789 (4)	0.7815 (11)	0.059 (14)*
N1	0.4310 (2)	0.3386 (3)	0.82838 (13)	0.0431 (7)
N2	0.3272 (2)	0.2283 (3)	0.72746 (13)	0.0401 (6)
O1	0.48006 (19)	0.0189 (3)	0.68755 (11)	0.0412 (6)
O2	0.5000	0.7436 (5)	0.7500	0.0605 (11)*
S1	0.19385 (15)	-0.05824 (19)	0.48627 (6)	0.0990 (6)
H1	0.1107	0.0061	0.4937	0.148*
S2	0.56823 (10)	-0.16647 (13)	0.59645 (5)	0.0678 (4)
H2	0.5567	-0.2860	0.6159	0.102*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C3	0.0367 (18)	0.0393 (18)	0.049 (2)	-0.0001 (13)	-0.0046 (14)	0.0043 (14)
C4	0.0496 (19)	0.0308 (15)	0.0336 (16)	-0.0073 (14)	-0.0017 (14)	0.0007 (12)
C5	0.0439 (18)	0.0357 (16)	0.0391 (17)	-0.0069 (14)	-0.0053 (14)	0.0024 (13)
C6	0.062 (2)	0.0407 (18)	0.0372 (17)	-0.0006 (16)	0.0042 (16)	-0.0029 (14)
C7	0.055 (2)	0.054 (2)	0.048 (2)	-0.0105 (18)	-0.0152 (17)	0.0112 (16)
C8	0.042 (2)	0.055 (2)	0.056 (2)	0.0115 (16)	0.0015 (17)	-0.0060 (17)
C9	0.086 (3)	0.053 (2)	0.0371 (19)	-0.012 (2)	-0.0050 (19)	-0.0067 (16)
C10	0.085 (3)	0.061 (2)	0.041 (2)	-0.021 (2)	-0.022 (2)	0.0033 (18)
C11	0.057 (2)	0.0418 (19)	0.055 (2)	0.0080 (17)	0.0037 (18)	-0.0054 (16)
C14	0.056 (3)	0.068 (3)	0.078 (3)	-0.004 (2)	0.003 (2)	-0.028 (2)
C15	0.076 (3)	0.066 (3)	0.054 (2)	0.011 (2)	0.018 (2)	0.004 (2)

supplementary materials

Co1	0.0383 (4)	0.0381 (4)	0.0406 (4)	0.000	-0.0028 (2)	0.000
N1	0.0433 (16)	0.0398 (15)	0.0462 (16)	0.0012 (12)	0.0001 (13)	-0.0065 (12)
N2	0.0376 (15)	0.0375 (14)	0.0452 (16)	0.0029 (11)	-0.0013 (12)	-0.0018 (13)
O1	0.0498 (13)	0.0367 (12)	0.0372 (12)	0.0030 (10)	-0.0080 (10)	-0.0059 (10)
S1	0.1182 (13)	0.1170 (13)	0.0616 (8)	-0.0154 (9)	-0.0498 (8)	-0.0119 (7)
S2	0.0758 (8)	0.0646 (7)	0.0629 (7)	0.0195 (6)	0.0044 (5)	-0.0196 (5)

Geometric parameters (\AA , $^\circ$)

C3—N2	1.267 (5)	C11—H11A	0.9700
C3—C5	1.449 (5)	C11—H11B	0.9700
C3—H3	0.9300	C14—N1	1.449 (5)
C4—O1	1.286 (4)	C14—H14A	0.9600
C4—C5	1.421 (5)	C14—H14B	0.9600
C4—C6	1.427 (5)	C14—H14C	0.9600
C5—C7	1.406 (5)	C15—N1	1.464 (5)
C6—C9	1.366 (5)	C15—H15A	0.9600
C6—S2	1.738 (4)	C15—H15B	0.9600
C7—C10	1.356 (6)	C15—H15C	0.9600
C7—H7	0.9300	Co1—O1	2.089 (2)
C8—N2	1.477 (5)	Co1—O1 ⁱ	2.089 (2)
C8—C11	1.505 (6)	Co1—N2 ⁱ	2.241 (3)
C8—H8A	0.9700	Co1—N2	2.241 (3)
C8—H8B	0.9700	Co1—N1	2.456 (3)
C9—C10	1.389 (7)	Co1—N1 ⁱ	2.456 (3)
C9—H9	0.9300	O2—H2A	0.832 (10)
C10—S1	1.735 (4)	S1—H1	1.2000
C11—N1	1.478 (5)	S2—H2	1.2000
N2—C3—C5	127.4 (3)	H11A—C11—H11B	107.9
N2—C3—H3	116.3	N1—C14—H14A	109.5
C5—C3—H3	116.3	N1—C14—H14B	109.5
O1—C4—C5	124.4 (3)	H14A—C14—H14B	109.5
O1—C4—C6	120.4 (3)	N1—C14—H14C	109.5
C5—C4—C6	115.2 (3)	H14A—C14—H14C	109.5
C7—C5—C4	120.2 (3)	H14B—C14—H14C	109.5
C7—C5—C3	116.8 (3)	N1—C15—H15A	109.5
C4—C5—C3	123.0 (3)	N1—C15—H15B	109.5
C9—C6—C4	123.6 (4)	H15A—C15—H15B	109.5
C9—C6—S2	119.1 (3)	N1—C15—H15C	109.5
C4—C6—S2	117.3 (3)	H15A—C15—H15C	109.5
C10—C7—C5	121.4 (4)	H15B—C15—H15C	109.5
C10—C7—H7	119.3	O1—Co1—O1 ⁱ	87.65 (14)
C5—C7—H7	119.3	O1—Co1—N2 ⁱ	114.06 (10)
N2—C8—C11	110.2 (3)	O1 ⁱ —Co1—N2 ⁱ	82.47 (10)
N2—C8—H8A	109.6	O1—Co1—N2	82.48 (10)
C11—C8—H8A	109.6	O1 ⁱ —Co1—N2	114.06 (10)
N2—C8—H8B	109.6	N2 ⁱ —Co1—N2	157.88 (15)

C11—C8—H8B	109.6	N1—Co1—O1	151.87 (12)
H8A—C8—H8B	108.1	N1—Co1—N2	73.88 (12)
C6—C9—C10	119.0 (4)	N1—Co1—N1 ⁱ	107.64 (12)
C6—C9—H9	120.5	C14—N1—C15	109.7 (4)
C10—C9—H9	120.5	C14—N1—C11	108.4 (3)
C7—C10—C9	120.4 (4)	C15—N1—C11	110.0 (3)
C7—C10—S1	120.7 (4)	C3—N2—C8	116.5 (3)
C9—C10—S1	118.9 (3)	C3—N2—Co1	126.0 (2)
N1—C11—C8	111.9 (3)	C8—N2—Co1	117.3 (2)
N1—C11—H11A	109.2	C4—O1—Co1	130.8 (2)
C8—C11—H11A	109.2	C10—S1—H1	109.5
N1—C11—H11B	109.2	C6—S2—H2	109.5
C8—C11—H11B	109.2		

Symmetry codes: (i) $-x+1, y, -z+3/2$.

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O2—H2A \cdots S2 ⁱⁱ	0.832 (10)	2.95 (2)	3.7001 (17)	151 (4)
O2—H2A \cdots O1 ⁱⁱ	0.832 (10)	2.25 (3)	2.928 (5)	139 (4)

Symmetry codes: (ii) $-x+1, y+1, -z+3/2$.

Fig. 1

