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

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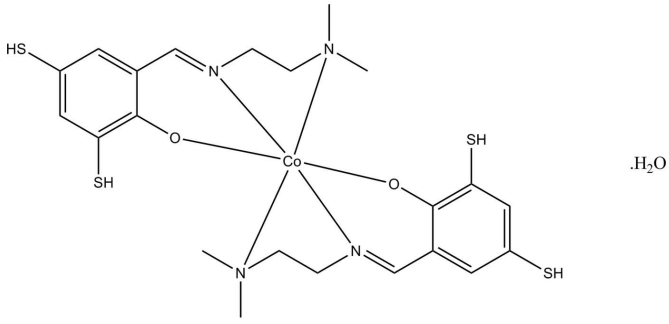
Key indicators: single-crystal X-ray study; T = 296 K; mean σ(C–C) = 0.005 Å; R factor = 0.054; wR factor = 0.186; data-to-parameter ratio = 15.9.

In the title hydrated complex, $[Co(C_{11}H_{15}N_2OS_2)_2]\cdot H_2O$, the Co^{II} atom (site symmetry 2) is coordinated by two *O*,*N*,*N*′-tridentate Schiff base ligands, resulting in a very distorted *cis*-CoO_2N_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 O—H···(O,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).



.H_2O

Experimental

Crystal data

$[Co(C_{11}H_{15}N_2OS_2)_2]\cdot H_2O$
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) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.01 mm^{-1}
T = 296 K
0.30 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Absorption correction: ψ scan (North *et al.*, 1968)
T_{min} = 0.752, T_{max} = 0.787
13847 measured reflections
2548 independent reflections
2039 reflections with $I > 2\sigma(I)$
R_{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
$\Delta\rho_{max}$ = 0.84 e Å^{-3}
$\Delta\rho_{min}$ = −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···A	D—H	H···A	D···A	D—H···A
O2—H2A···S2^{i}	0.832 (10)	2.95 (2)	3.7001 (17)	151 (4)
O2—H2A···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*.

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

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supporting information

Acta Cryst. (2009). E**65**, m1146 [doi:10.1107/S1600536809033650]

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

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S1. Comment

As part of our onging 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 octhaedral coordination by two N,N,O-tridentate Schiff base ligands.

S2. Experimental

A mixture of 2-hydroxy-3,5-dimercaptobenzaldehyde (372 mg, 2 mmol), *N*,*N*-dimethylethane-1,2-diamine (176 mg, 2 mmol) and $CoCl_2.6H_2O$ (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.

S3. 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 = 0.93–0.97Å, S—H = 1.2Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(carrier) or 1.5U_{eq}(methyl C).

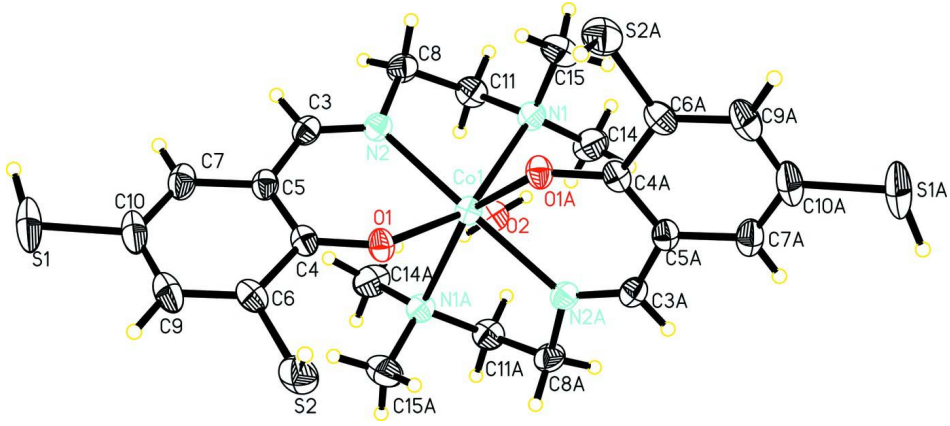

Figure 1

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).

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Crystal data

$[Co(C_{11}H_{15}N_2OS_2)_2]\cdot H_2O$
$M_r = 587.69$
Orthorhombic, *Pbcn*
Hall symbol: -P 2n 2ab
$a = 12.3755\ (15)$ Å
$b = 9.2485\ (15)$ Å
$c = 22.827\ (3)$ Å
$V = 2612.7\ (6)$ Å^3
$Z = 4$
$F(000) = 1228$
$D_x = 1.494$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 9$–$12°$
$\mu = 1.01$ mm^{-1}
$T = 296$ K
Block, red
0.30 × 0.25 × 0.25 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
$\omega/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.752$, $T_{max} = 0.787$
13847 measured reflections
2548 independent reflections
2039 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 26.0°$, $\theta_{min} = 2.4°$
$h = -15\rightarrow15$
$k = -11\rightarrow6$
$l = -28\rightarrow27$
200 standard reflections every 3 reflections
intensity decay: 1%

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.054$
$wR(F^2) = 0.186$
$S = 1.06$
2548 reflections
160 parameters
1 restraint
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1201P)^2 + 2.1692P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.84$ e Å^{-3}
$\Delta\rho_{min} = -0.96$ 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}
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 (Å²)

	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)
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 (Å, º)

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

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O2—H2*A*···S2^{ii}	0.83 (1)	2.95 (2)	3.7001 (17)	151 (4)
O2—H2*A*···O1^{ii}	0.83 (1)	2.25 (3)	2.928 (5)	139 (4)

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