

Bis(pyridine-2-methanol- κ^2N,O]-bis(thiocyanato- κN)cobalt(II)

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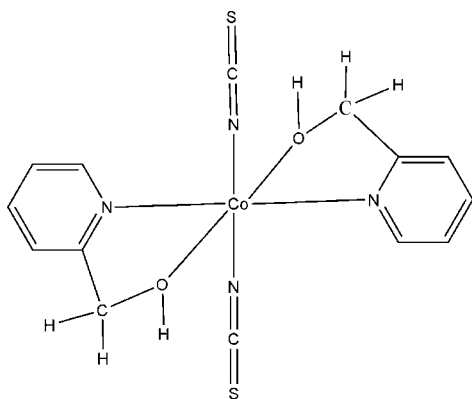
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(C-C) = 0.008$ Å; R factor = 0.055; wR factor = 0.170; data-to-parameter ratio = 13.9.

The Co^{II} ion in the title complex, $[Co(NCS)_2(C_6H_7NO)_2]$, lies on a crystallographic twofold axis and is in a slightly distorted octahedral coordination environment. In the crystal structure, intermolecular $O-H \cdots S$ hydrogen bonds link molecules to form a two-dimensional network perpendicular to the c axis.

Related literature

 For related literature, see: Yang *et al.* (2003); Yoo *et al.* (2000).


Experimental

Crystal data

 $[Co(NCS)_2(C_6H_7NO)_2]$
 $M_r = 393.34$

 Orthorhombic, $Pbcn$
 $a = 11.371$ (10) Å

 $b = 8.255$ (8) Å

 $c = 17.779$ (16) Å

 $V = 1669$ (3) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 1.29$ mm⁻¹
 $T = 298$ (2) K

 $0.45 \times 0.37 \times 0.30$ mm

Data collection

Bruker SMART CCD diffractometer

Absorption correction: multi-scan (SADABS; Sheldrick, 1996)

 $T_{min} = 0.594$, $T_{max} = 0.698$

6193 measured reflections

1463 independent reflections

 1079 reflections with $I > 2\sigma(I)$
 $R_{int} = 0.088$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.055$
 $wR(F^2) = 0.170$
 $S = 1.00$

1463 reflections

105 parameters

H-atom parameters constrained

 $\Delta\rho_{max} = 0.90$ e Å⁻³
 $\Delta\rho_{min} = -0.80$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

Co1—N2	2.039 (5)	Co1—O1	2.239 (4)
Co1—N1	2.093 (4)		
N2 ⁱ —Co1—N2	97.4 (2)	N1—Co1—O1 ⁱ	86.92 (15)
N2—Co1—N1	97.81 (17)	N2—Co1—O1	172.34 (15)
N2—Co1—N1 ⁱ	99.78 (17)	N1—Co1—O1	74.63 (15)
N1—Co1—N1 ⁱ	153.2 (2)	O1 ⁱ —Co1—O1	93.0 (2)
N2—Co1—O1 ⁱ	85.30 (16)		

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

Table 2

Hydrogen-bond geometry (Å, °).

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O1—H1 ⁱ \cdots S1 ⁱⁱ	0.82	2.51	3.296 (5)	161

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

Data collection: SMART (Siemens, 1996); cell refinement: SAINT (Siemens, 1996); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997a); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997a); molecular graphics: SHELXTL (Sheldrick, 1997b); 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: LH2553).

References

- Sheldrick, G. M. (1996). *SADABS*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997a). *SHELXL97* and *SHELXS97*. University of Göttingen, Germany.
- Sheldrick, G. M. (1997b). *SHELXTL*. Version 5.1. Bruker AXS Inc., Madison, Wisconsin, USA.
- Siemens (1996). *SMART* and *SAINTE*. Siemens Analytical X-ray Instruments Inc., Madison, Wisconsin, USA.
- Yang, E.-C., Harden, N., Wernsdorfer, W., Zakharov, L., Brechin, E. K., Rheingold, A. L., Christou, G. & Hendrickson, D. N. (2003). *Polyhedron*, **22**, 1857–1863.
- Yoo, J., Brechin, E. K., Yamaguchi, A., Nakano, M., Huffman, J. C., Maniero, A. L., Brunel, L.-C., Awaga, K., Ishimoto, H., Christou, G. & Hendrickson, D. N. (2000). *Inorg. Chem.* **39**, 3615–3623.

supplementary materials

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Bis(pyridine-2-methanol- κ^2N,O)bis(thiocyanato- κN)cobalt(II)

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Comment

In recent years there has been considerable interest in metal complexes of hydroxymethyl-pyridine due to the ligands versatile coordination modes and bridging function (Yoo *et al.*, 2000; Yang *et al.*, 2003). As an extension of this work on the structural characterization of hydroxymethyl-pyridine derivatives, we have synthesized here title compound, (I), and report herein its crystal structure.

The complex (Fig. 1) consists of two L^{2-} (L =(hydroxymethyl)(pyridine) ligands, one Co^{II} ion and two thiocyanato ligands. The coordination geometry around the Co center is slightly distorted octahedral with a CoN_4O_2 ligand set (see table of geometric parameters). Atom N2 of a thiocyanato ligand and atom O1 of an hydroxymethylpyridine ligand occupy the axial sites. In the crystal structure, intermolecular $\text{O}\cdots\text{S}$ hydrogen bonds link molecules to form a two-dimensional network perpendicular to the c axis (see hydrogen-bond geometry table).

Experimental

2-hydroxymethylpyridine (0.546 g, 5 mmol) and Et_4NOH with 20% aqueous solution (0.736 g, 1 mmol) were added to a mixture (30 ml) of methanol and acetonitrile ($V_1:V_2=2:1$) containing cobaltous chlorine hexahydrate (0.476 g, 2 mmol). After the solution was stirred at room temperature for 0.5 h. Potassium thiocyanato (0.486 g, 5 mmol) in 5 ml of methanol was added to the above solution and then further stirred for 2 h. The resulting clear solution was filtered and left to stand at room temperature. Red crystal suitable for X-ray diffraction were obtained by slow evaporation of the solvents within 2 weeks. (m.p.=458 K-459 K). Elemental analysis calculated for $\text{C}_{14}\text{H}_{14}\text{N}_4\text{O}_2\text{CoS}_2$: C 42.75, H 3.59, N 14.24%; found: C 42.80, H 3.54, N 14.30%.

Refinement

All H atoms were placed geometrically and treated as riding on their parent atoms with C—H 0.93(pyridine), C—H 0.97 (methylene) Å [$U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$] and O—H 0.82 Å (hydroxyl) [$U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{O})$].

Figures

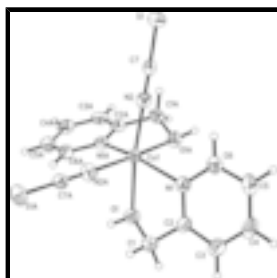


Fig. 1. The molecule structure of the title complex, showing 30% probability displacement ellipsoids and the atom-numbering scheme. Atoms labelled with the suffix A are generated by the symmetry operation $(-x + 1, y, -z + 1/2)$. H atoms have been omitted for clarity.

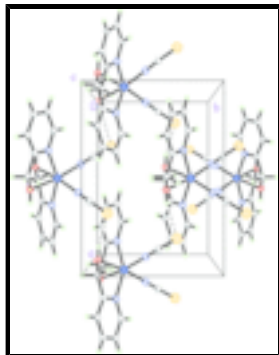


Fig. 2. Part of the crystal structure with hydrogen bonds shown as dashed lines.

Bis(pyridine-2-methanol- κ^2N,O)]bis(thiocyanato- κN)cobalt(II)

Crystal data

[Co(NCS)₂(C₆H₇NO)₂]

$M_r = 393.34$

Orthorhombic, *Pbcn*

Hall symbol: -P 2n 2ab

$a = 11.371 (10) \text{ \AA}$

$b = 8.255 (8) \text{ \AA}$

$c = 17.779 (16) \text{ \AA}$

$V = 1669 (3) \text{ \AA}^3$

$Z = 4$

$F_{000} = 804$

$D_x = 1.565 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation

$\lambda = 0.71073 \text{ \AA}$

Cell parameters from 2060 reflections

$\theta = 2.9\text{--}24.0^\circ$

$\mu = 1.29 \text{ mm}^{-1}$

$T = 298 (2) \text{ K}$

Block, red

$0.45 \times 0.37 \times 0.30 \text{ mm}$

Data collection

Bruker SMART CCD
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$T = 298(2) \text{ K}$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 1996)

$T_{\min} = 0.594$, $T_{\max} = 0.698$

6193 measured reflections

1463 independent reflections

1079 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.088$

$\theta_{\max} = 25.0^\circ$

$\theta_{\min} = 2.3^\circ$

$h = -12 \rightarrow 13$

$k = -8 \rightarrow 9$

$l = -18 \rightarrow 20$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.055$

$wR(F^2) = 0.170$

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0972P)^2 + 2.6034P]$

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

$S = 1.00$	$(\Delta/\sigma)_{\max} < 0.001$
1463 reflections	$\Delta\rho_{\max} = 0.90 \text{ e } \text{\AA}^{-3}$
105 parameters	$\Delta\rho_{\min} = -0.80 \text{ e } \text{\AA}^{-3}$
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	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.5000	0.21568 (11)	0.2500	0.0366 (4)
N1	0.6485 (4)	0.2744 (5)	0.1860 (2)	0.0415 (10)
N2	0.5778 (4)	0.0526 (5)	0.3203 (2)	0.0490 (11)
O1	0.4388 (3)	0.4024 (4)	0.16747 (19)	0.0506 (9)
H1	0.3691	0.3971	0.1558	0.076*
S1	0.68424 (13)	-0.17410 (19)	0.41381 (8)	0.0573 (5)
C1	0.5087 (4)	0.4002 (8)	0.1015 (3)	0.0516 (14)
H1A	0.5070	0.5057	0.0776	0.062*
H1B	0.4779	0.3211	0.0663	0.062*
C2	0.6329 (4)	0.3572 (6)	0.1224 (3)	0.0431 (12)
C3	0.7262 (5)	0.4016 (7)	0.0772 (3)	0.0576 (15)
H3	0.7133	0.4607	0.0334	0.069*
C4	0.8373 (5)	0.3579 (8)	0.0975 (3)	0.0593 (16)
H4	0.9008	0.3835	0.0667	0.071*
C5	0.8549 (5)	0.2756 (7)	0.1639 (4)	0.0592 (16)
H5	0.9304	0.2491	0.1799	0.071*
C6	0.7586 (5)	0.2337 (6)	0.2058 (3)	0.0482 (13)
H6	0.7699	0.1746	0.2498	0.058*
C7	0.6233 (4)	-0.0404 (6)	0.3589 (3)	0.0392 (11)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0326 (5)	0.0353 (6)	0.0419 (6)	0.000	0.0008 (4)	0.000
N1	0.045 (2)	0.043 (2)	0.036 (2)	-0.0052 (18)	0.0000 (18)	-0.0067 (19)
N2	0.047 (3)	0.043 (2)	0.057 (3)	-0.004 (2)	-0.003 (2)	0.008 (2)
O1	0.047 (2)	0.054 (2)	0.051 (2)	0.0069 (17)	-0.0007 (17)	0.0056 (17)

supplementary materials

S1	0.0566 (9)	0.0564 (9)	0.0590 (9)	0.0030 (7)	-0.0096 (7)	0.0154 (7)
C1	0.053 (3)	0.062 (4)	0.039 (3)	-0.005 (3)	-0.004 (2)	0.004 (3)
C2	0.050 (3)	0.043 (3)	0.037 (3)	-0.010 (2)	0.004 (2)	-0.005 (2)
C3	0.071 (4)	0.062 (3)	0.040 (3)	-0.020 (3)	0.004 (3)	-0.009 (3)
C4	0.052 (3)	0.074 (4)	0.052 (4)	-0.014 (3)	0.021 (3)	-0.014 (3)
C5	0.038 (3)	0.072 (4)	0.067 (4)	-0.007 (3)	0.000 (3)	-0.016 (3)
C6	0.044 (3)	0.045 (3)	0.056 (3)	-0.005 (2)	-0.002 (2)	-0.004 (2)
C7	0.034 (2)	0.039 (3)	0.045 (3)	-0.008 (2)	-0.001 (2)	-0.004 (2)

Geometric parameters (Å, °)

Co1—N2 ⁱ	2.039 (5)	C1—C2	1.502 (7)
Co1—N2	2.039 (5)	C1—H1A	0.9700
Co1—N1	2.093 (4)	C1—H1B	0.9700
Co1—N1 ⁱ	2.093 (4)	C2—C3	1.381 (7)
Co1—O1 ⁱ	2.239 (4)	C3—C4	1.363 (8)
Co1—O1	2.239 (4)	C3—H3	0.9300
N1—C2	1.333 (6)	C4—C5	1.377 (9)
N1—C6	1.344 (7)	C4—H4	0.9300
N2—C7	1.152 (6)	C5—C6	1.369 (8)
O1—C1	1.417 (6)	C5—H5	0.9300
O1—H1	0.8200	C6—H6	0.9300
S1—C7	1.629 (5)		
N2 ⁱ —Co1—N2	97.4 (2)	O1—C1—C2	109.0 (4)
N2 ⁱ —Co1—N1	99.78 (17)	O1—C1—H1A	109.9
N2—Co1—N1	97.81 (17)	C2—C1—H1A	109.9
N2 ⁱ —Co1—N1 ⁱ	97.81 (17)	O1—C1—H1B	109.9
N2—Co1—N1 ⁱ	99.78 (17)	C2—C1—H1B	109.9
N1—Co1—N1 ⁱ	153.2 (2)	H1A—C1—H1B	108.3
N2 ⁱ —Co1—O1 ⁱ	172.34 (15)	N1—C2—C3	121.9 (5)
N2—Co1—O1 ⁱ	85.30 (16)	N1—C2—C1	117.1 (4)
N1—Co1—O1 ⁱ	86.92 (15)	C3—C2—C1	121.0 (5)
N1 ⁱ —Co1—O1 ⁱ	74.63 (15)	C4—C3—C2	119.2 (5)
N2 ⁱ —Co1—O1	85.30 (16)	C4—C3—H3	120.4
N2—Co1—O1	172.34 (15)	C2—C3—H3	120.4
N1—Co1—O1	74.63 (15)	C3—C4—C5	119.5 (5)
N1 ⁱ —Co1—O1	86.92 (15)	C3—C4—H4	120.2
O1 ⁱ —Co1—O1	93.0 (2)	C5—C4—H4	120.2
C2—N1—C6	118.3 (4)	C6—C5—C4	118.3 (5)
C2—N1—Co1	118.2 (3)	C6—C5—H5	120.8
C6—N1—Co1	123.4 (4)	C4—C5—H5	120.8
C7—N2—Co1	178.7 (4)	N1—C6—C5	122.7 (5)
C1—O1—Co1	111.0 (3)	N1—C6—H6	118.7
C1—O1—H1	109.4	C5—C6—H6	118.7
Co1—O1—H1	115.4	N2—C7—S1	178.5 (4)

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

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
O1—H1 \cdots S1 ⁱⁱ	0.82	2.51	3.296 (5)	161

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

Fig. 2

