

{Bis[4-(2-pyridyl)pyrimidin-2-yl]sulfane}-dichloridocobalt(II)

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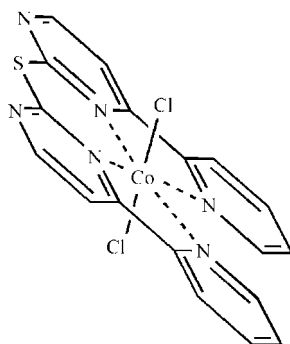
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 Key indicators: single-crystal X-ray study; $T = 298$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.046; wR factor = 0.077; data-to-parameter ratio = 18.0.

The asymmetric unit of the title compound, $[\text{CoCl}_2(\text{C}_{18}\text{H}_{12}\text{N}_6\text{S})]$, contains one half-molecule situated on a twofold rotation axis which passes through the Co and S atoms. The metal centre is in a distorted octahedral CoCl_2N_4 coordination with the Cl atoms in the axial positions. In the crystal structure, intermolecular $\text{C}-\text{H}\cdots\text{Cl}$ interactions help to establish the packing.

Related literature

For coordination compounds with bis(4-pyridinyl)sulfane, see: Jung *et al.* (1998); Ni & Vittal (2001).



Experimental

Crystal data

 $[\text{CoCl}_2(\text{C}_{18}\text{H}_{12}\text{N}_6\text{S})]$
 $M_r = 474.24$

 Monoclinic, $C2/c$
 $a = 14.685$ (3) Å
 $b = 10.325$ (2) Å
 $c = 13.376$ (3) Å
 $\beta = 112.339$ (3)°
 $V = 1875.9$ (7) Å³
 $Z = 4$
 Mo $K\alpha$ radiation
 $\mu = 1.33$ mm⁻¹
 $T = 298$ K
 $0.20 \times 0.18 \times 0.12$ mm

Data collection

 Bruker APEXII CCD area-detector diffractometer
 Absorption correction: multi-scan (SADABS; Bruker, 2001)
 $T_{\min} = 0.884$, $T_{\max} = 0.920$
 (expected range = 0.819–0.853)

 6015 measured reflections
 2302 independent reflections
 1526 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.141$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.046$
 $wR(F^2) = 0.077$
 $S = 1.00$
 2302 reflections

 128 parameters
 H-atom parameters constrained
 $\Delta\rho_{\max} = 0.51$ e Å⁻³
 $\Delta\rho_{\min} = -0.68$ e Å⁻³
Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{C}2-\text{H}2A\cdots\text{Cl}1^{\text{i}}$	0.93	2.63	3.546 (3)	170
$\text{C}3-\text{H}3A\cdots\text{Cl}1^{\text{ii}}$	0.93	2.73	3.584 (3)	154
$\text{C}7-\text{H}7A\cdots\text{Cl}1^{\text{iii}}$	0.93	2.76	3.580 (4)	148

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

Data collection: APEX2 (Bruker, 2007); cell refinement: SAINT-Plus (Bruker, 2007); data reduction: SAINT-Plus; 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: AT2809).

References

- Bruker (2001). SADABS. Bruker AXS Inc., Madison, Wisconsin, USA.
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 Jung, O. S., Park, S. H., Kim, D. C. & Kim, K. M. (1998). *Inorg. Chem.* **37**, 610–611.
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supplementary materials

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{Bis[4-(2-pyridyl)pyrimidin-2-yl]sulfane}dichloridocobalt(II)

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Comment

Organic ligand of bis(4-pyridinyl)sulfane has been employed to construct some intriguing metal–organic frameworks (MOFs) (Jung *et al.*, 1998; Ni & Vittal, 2001). Herein, we report the molecular structure of a mononuclear Co^{II} coordination complex (I) with bis(4-(pyridin-2-yl)pyrimidin-2-yl)sulfane.

In compound (I), the cobalt(II) ion is six-coordinated by four N atoms in equatorial position and two Cl atoms in apical position (Fig. 1). The Co—N bond lengths are 2.102 (2) Å and 2.130 (2) Å and the Co—Cl bond distance is 2.4363 (9) Å. In the crystal, intermolecular C—H⋯Cl interactions help to establish the packing.

Experimental

To a solution of CoCl₂ (0.1 mmol) in MeOH (10 ml) was added a solution of bis(4-(pyridin-2-yl)pyrimidin-2-yl)sulfane (0.1 mmol) in CH₂Cl₂ (5 ml). The mixture was stirred for 30 min, then filtered. The mother liquid was stood at ambient temperature for two days to give the orange crystals.

Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 Å and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for aromatic H atoms.

Figures

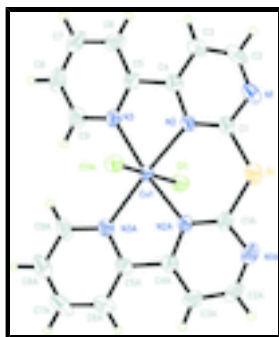


Fig. 1. The molecular structure of the title compound showing the atomic numbering and 40% probability displacement ellipsoids [symmetry code: (A) $-x, y, 1/2 - z$.]

{Bis[4-(2-pyridyl)pyrimidin-2-yl]sulfane}dichloridocobalt(II)

Crystal data

[CoCl₂(C₁₈H₁₂N₆S)]

$F_{000} = 956$

supplementary materials

$$M_r = 474.24$$

Monoclinic, $C2/c$

Hall symbol: $-C\ 2yc$

$$a = 14.685\ (3)\ \text{\AA}$$

$$b = 10.325\ (2)\ \text{\AA}$$

$$c = 13.376\ (3)\ \text{\AA}$$

$$\beta = 112.339\ (3)^\circ$$

$$V = 1875.9\ (7)\ \text{\AA}^3$$

$$Z = 4$$

$$D_x = 1.679\ \text{Mg m}^{-3}$$

Mo $K\alpha$ radiation

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

Cell parameters from 2302 reflections

$$\theta = 2.3\text{--}25.5^\circ$$

$$\mu = 1.33\ \text{mm}^{-1}$$

$$T = 298\ \text{K}$$

Block, orange

$$0.20 \times 0.18 \times 0.12\ \text{mm}$$

Data collection

Bruker APEXII CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Monochromator: graphite

$$T = 298\ \text{K}$$

φ and ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2001)

$$T_{\min} = 0.884, T_{\max} = 0.920$$

6015 measured reflections

2302 independent reflections

1526 reflections with $I > 2\sigma(I)$

$$R_{\text{int}} = 0.141$$

$$\theta_{\max} = 28.4^\circ$$

$$\theta_{\min} = 2.5^\circ$$

$$h = -17 \rightarrow 19$$

$$k = -6 \rightarrow 13$$

$$l = -17 \rightarrow 17$$

Refinement

Refinement on F^2

Least-squares matrix: full

$$R[F^2 > 2\sigma(F^2)] = 0.046$$

$$wR(F^2) = 0.077$$

$$S = 1.00$$

2302 reflections

128 parameters

Primary atom site location: structure-invariant direct
methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring
sites

H-atom parameters constrained

$$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$$

$$\text{where } P = (F_o^2 + 2F_c^2)/3$$

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

$$\Delta\rho_{\max} = 0.51\ \text{e \AA}^{-3}$$

$$\Delta\rho_{\min} = -0.67\ \text{e \AA}^{-3}$$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Co1	0.5000	0.21623 (4)	0.2500	0.03357 (15)
Cl1	0.52209 (5)	0.21891 (6)	0.07848 (5)	0.04298 (19)
S1	0.5000	0.57242 (8)	0.2500	0.0646 (4)
N3	0.37405 (17)	0.09651 (17)	0.17599 (17)	0.0358 (5)
N2	0.38470 (16)	0.35147 (16)	0.19288 (16)	0.0327 (5)
C4	0.2933 (2)	0.3022 (2)	0.15111 (19)	0.0353 (6)
C1	0.3909 (2)	0.4812 (2)	0.2002 (2)	0.0382 (7)
N1	0.3163 (2)	0.56366 (19)	0.16953 (19)	0.0473 (6)
C5	0.2875 (2)	0.1579 (2)	0.14019 (19)	0.0350 (6)
C3	0.2114 (2)	0.3803 (3)	0.1175 (2)	0.0456 (7)
H3A	0.1482	0.3460	0.0884	0.055*
C2	0.2275 (2)	0.5119 (3)	0.1289 (2)	0.0500 (8)
H2A	0.1733	0.5669	0.1070	0.060*
C6	0.1984 (2)	0.0954 (2)	0.0938 (2)	0.0472 (7)
H6A	0.1398	0.1419	0.0695	0.057*
C8	0.2854 (3)	-0.1011 (3)	0.1176 (2)	0.0586 (9)
H8A	0.2869	-0.1906	0.1108	0.070*
C9	0.3722 (2)	-0.0329 (2)	0.1618 (2)	0.0523 (9)
H9A	0.4315	-0.0777	0.1825	0.063*
C7	0.1980 (3)	-0.0383 (3)	0.0841 (2)	0.0566 (9)
H7A	0.1390	-0.0837	0.0552	0.068*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Co1	0.0269 (3)	0.0223 (2)	0.0436 (3)	0.000	0.0045 (2)	0.000
Cl1	0.0342 (4)	0.0443 (4)	0.0449 (4)	0.0018 (3)	0.0089 (3)	0.0000 (3)
S1	0.0532 (8)	0.0242 (5)	0.0984 (10)	0.000	0.0086 (7)	0.000
N3	0.0360 (14)	0.0272 (10)	0.0381 (12)	-0.0012 (10)	0.0072 (10)	0.0015 (9)
N2	0.0333 (13)	0.0253 (10)	0.0349 (12)	0.0029 (10)	0.0077 (10)	0.0012 (8)
C4	0.0382 (17)	0.0384 (14)	0.0244 (13)	0.0011 (13)	0.0064 (12)	0.0006 (10)
C1	0.0434 (18)	0.0290 (12)	0.0390 (15)	0.0063 (12)	0.0121 (14)	0.0017 (11)
N1	0.0512 (18)	0.0374 (12)	0.0472 (15)	0.0170 (12)	0.0117 (13)	0.0037 (10)
C5	0.0354 (16)	0.0382 (13)	0.0280 (13)	-0.0061 (13)	0.0083 (13)	0.0016 (11)
C3	0.0343 (17)	0.0556 (17)	0.0403 (16)	0.0079 (15)	0.0067 (14)	0.0014 (13)
C2	0.047 (2)	0.0529 (17)	0.0445 (18)	0.0276 (16)	0.0118 (16)	0.0072 (13)
C6	0.0395 (18)	0.0564 (17)	0.0403 (16)	-0.0103 (15)	0.0092 (14)	0.0023 (13)
C8	0.076 (3)	0.0356 (14)	0.0502 (19)	-0.0182 (17)	0.0082 (19)	-0.0005 (13)
C9	0.058 (2)	0.0286 (13)	0.0580 (19)	-0.0056 (14)	0.0079 (17)	-0.0030 (12)
C7	0.059 (2)	0.0570 (18)	0.0466 (18)	-0.0322 (17)	0.0116 (17)	-0.0023 (14)

supplementary materials

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

Co1—N2 ⁱ	2.102 (2)	C1—N1	1.323 (3)
Co1—N2	2.1017 (19)	N1—C2	1.320 (4)
Co1—N3	2.130 (2)	C5—C6	1.377 (4)
Co1—N3 ⁱ	2.130 (2)	C3—C2	1.378 (3)
Co1—Cl1 ⁱ	2.4363 (9)	C3—H3A	0.9300
Co1—Cl1	2.4363 (9)	C2—H2A	0.9300
S1—C1	1.757 (3)	C6—C7	1.386 (3)
S1—C1 ⁱ	1.757 (3)	C6—H6A	0.9300
N3—C5	1.335 (3)	C8—C7	1.354 (5)
N3—C9	1.349 (3)	C8—C9	1.377 (4)
N2—C4	1.343 (3)	C8—H8A	0.9300
N2—C1	1.343 (3)	C9—H9A	0.9300
C4—C3	1.374 (4)	C7—H7A	0.9300
C4—C5	1.497 (3)		
N2 ⁱ —Co1—N2	96.73 (11)	N1—C1—N2	126.5 (3)
N2 ⁱ —Co1—N3	172.65 (7)	N1—C1—S1	107.47 (18)
N2—Co1—N3	77.25 (8)	N2—C1—S1	126.1 (2)
N2 ⁱ —Co1—N3 ⁱ	77.25 (8)	C2—N1—C1	116.0 (2)
N2—Co1—N3 ⁱ	172.65 (7)	N3—C5—C6	123.5 (2)
N3—Co1—N3 ⁱ	109.05 (11)	N3—C5—C4	115.3 (2)
N2 ⁱ —Co1—Cl1 ⁱ	91.57 (6)	C6—C5—C4	121.3 (3)
N2—Co1—Cl1 ⁱ	87.57 (6)	C4—C3—C2	116.8 (3)
N3—Co1—Cl1 ⁱ	92.37 (6)	C4—C3—H3A	121.6
N3 ⁱ —Co1—Cl1 ⁱ	88.39 (6)	C2—C3—H3A	121.6
N2 ⁱ —Co1—Cl1	87.57 (6)	N1—C2—C3	123.1 (3)
N2—Co1—Cl1	91.57 (6)	N1—C2—H2A	118.5
N3—Co1—Cl1	88.39 (6)	C3—C2—H2A	118.5
N3 ⁱ —Co1—Cl1	92.37 (6)	C5—C6—C7	118.6 (3)
Cl1 ⁱ —Co1—Cl1	178.70 (3)	C5—C6—H6A	120.7
C1—S1—C1 ⁱ	115.15 (17)	C7—C6—H6A	120.7
C5—N3—C9	117.0 (2)	C7—C8—C9	120.3 (3)
C5—N3—Co1	115.55 (15)	C7—C8—H8A	119.9
C9—N3—Co1	127.4 (2)	C9—C8—H8A	119.9
C4—N2—C1	115.9 (2)	N3—C9—C8	122.1 (3)
C4—N2—Co1	116.07 (15)	N3—C9—H9A	118.9
C1—N2—Co1	127.82 (19)	C8—C9—H9A	118.9
N2—C4—C3	121.7 (2)	C8—C7—C6	118.4 (3)
N2—C4—C5	115.4 (2)	C8—C7—H7A	120.8
C3—C4—C5	122.9 (3)	C6—C7—H7A	120.8
N2—Co1—N3—C5	-5.56 (17)	C1 ⁱ —S1—C1—N1	177.9 (2)
N3 ⁱ —Co1—N3—C5	170.5 (2)	C1 ⁱ —S1—C1—N2	-3.15 (18)
Cl1 ⁱ —Co1—N3—C5	81.40 (18)	N2—C1—N1—C2	0.7 (4)

Cl1—Co1—N3—C5	-97.53 (18)	S1—C1—N1—C2	179.6 (2)
N2—Co1—N3—C9	174.6 (2)	C9—N3—C5—C6	2.2 (4)
N3 ⁱ —Co1—N3—C9	-9.30 (19)	Co1—N3—C5—C6	-177.6 (2)
Cl1 ⁱ —Co1—N3—C9	-98.4 (2)	C9—N3—C5—C4	-176.2 (2)
Cl1—Co1—N3—C9	82.7 (2)	Co1—N3—C5—C4	3.9 (3)
N2 ⁱ —Co1—N2—C4	-177.8 (2)	N2—C4—C5—N3	1.7 (3)
N3—Co1—N2—C4	6.51 (17)	C3—C4—C5—N3	179.4 (2)
Cl1 ⁱ —Co1—N2—C4	-86.47 (17)	N2—C4—C5—C6	-176.9 (2)
Cl1—Co1—N2—C4	94.50 (17)	C3—C4—C5—C6	0.9 (4)
N2 ⁱ —Co1—N2—C1	-3.04 (17)	N2—C4—C3—C2	-0.2 (4)
N3—Co1—N2—C1	-178.8 (2)	C5—C4—C3—C2	-177.8 (2)
Cl1 ⁱ —Co1—N2—C1	88.2 (2)	C1—N1—C2—C3	-0.5 (4)
Cl1—Co1—N2—C1	-90.8 (2)	C4—C3—C2—N1	0.2 (4)
C1—N2—C4—C3	0.3 (4)	N3—C5—C6—C7	0.4 (4)
Co1—N2—C4—C3	175.7 (2)	C4—C5—C6—C7	178.8 (2)
C1—N2—C4—C5	178.1 (2)	C5—N3—C9—C8	-3.3 (4)
Co1—N2—C4—C5	-6.5 (3)	Co1—N3—C9—C8	176.5 (2)
C4—N2—C1—N1	-0.6 (4)	C7—C8—C9—N3	1.7 (5)
Co1—N2—C1—N1	-175.4 (2)	C9—C8—C7—C6	1.0 (5)
C4—N2—C1—S1	-179.38 (19)	C5—C6—C7—C8	-2.0 (4)
Co1—N2—C1—S1	5.9 (3)		

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

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

<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>
C2—H2A \cdots Cl1 ⁱⁱ	0.93	2.63	3.546 (3)	170
C3—H3A \cdots Cl1 ⁱⁱⁱ	0.93	2.73	3.584 (3)	154
C7—H7A \cdots Cl1 ^{iv}	0.93	2.76	3.580 (4)	148

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

Fig. 1

