metal-organic compounds

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

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The asymmetric unit of the title compound, [CoCl$_2$-(C$_{18}$H$_{12}$N$_6$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$_2$N$_4$ coordination with the Cl atoms in the axial positions. In the crystal structure, intermolecular C—H···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).
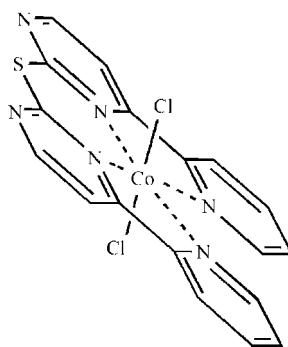
Experimental

Crystal data

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

Monoclinic, $C2/c$
a = 14.685 (3) Å
b = 10.325 (2) Å
c = 13.376 (3) Å
β = 112.339 (3)°
V = 1875.9 (7) Å^3

Z = 4
Mo $K\alpha$ radiation
μ = 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_{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—H···A	D—H	H···A	D···A	D—H···A
C2—H2A···Cl1^i	0.93	2.63	3.546 (3)	170
C3—H3A···Cl1^{ii}	0.93	2.73	3.584 (3)	154
C7—H7A···Cl1^{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.

References

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Bruker (2007). APEX2 and SAINT-Plus. Bruker AXS Inc., Madison, Wisconsin, USA.
Jung, O. S., Park, S. H., Kim, D. C. & Kim, K. M. (1998). Inorg. Chem. 37, 610-611.
Ni, Z. & Vittal, J. J. (2001). Cryst. Growth Des. 1, 195-197.
Sheldrick, G. M. (2008). Acta Cryst. A64, 112–122.

supporting information

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

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S1. Comment

Organic ligand of bis(4-pyridinyl)sulfane has been employed to construct some intriguing metal–orgainc 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.

S2. Experimental

To a solution of $CoCl_2$ (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 CH_2Cl_2 (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.

S3. Refinement

All H atoms were positioned geometrically and allowed to ride on their parent atoms, with C—H = 0.93 Å and U_{iso}(H) = 1.2U_{eq}(C) for aromatic H atoms.
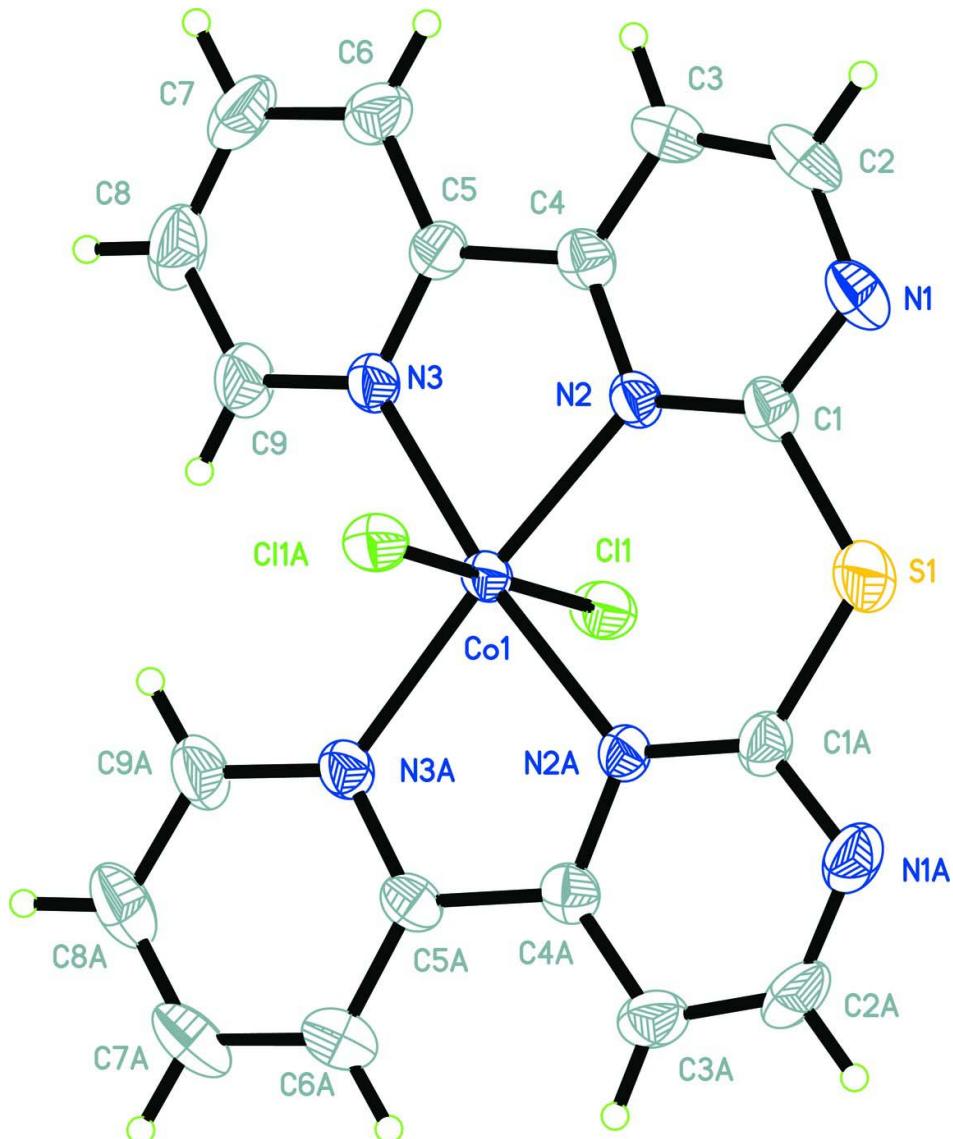
Figure 1

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

[CoCl$_2$(C$_{18}$H$_{12}$N$_6$S)]	$F(000) = 956$
$M_r = 474.24$	$D_x = 1.679$ Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Hall symbol: -C 2yc	Cell parameters from 2302 reflections
$a = 14.685$ (3) Å	$\theta = 2.3–25.5°$
$b = 10.325$ (2) Å	$\mu = 1.33$ mm^{-1}
$c = 13.376$ (3) Å	$T = 298$ K
$\beta = 112.339$ (3)°	Block, orange
$V = 1875.9$ (7) Å^3	$0.20 \times 0.18 \times 0.12$ mm
$Z = 4$	

Data collection

Bruker APEXII CCD area-detector diffractometer	6015 measured reflections
Radiation source: fine-focus sealed tube	2302 independent reflections
Graphite monochromator	1526 reflections with $I > 2\sigma(I)$
φ and ω scans	$R_{int} = 0.141$
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)	$\theta_{max} = 28.4°$, $\theta_{min} = 2.5°$
$T_{min} = 0.884$, $T_{max} = 0.920$	$h = -17\rightarrow19$
	$k = -6\rightarrow13$
	$l = -17\rightarrow17$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	
$R[F^2 > 2\sigma(F^2)] = 0.046$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.077$	
$S = 1.00$	H-atom parameters constrained
2302 reflections	$w = 1/[\sigma^2(F_o^2) + (0.01P)^2]$
128 parameters	where $P = (F_o^2 + 2F_c^2)/3$
0 restraints	$(\Delta/\sigma)_{max} = 0.001$
Primary atom site location: structure-invariant direct methods	$\Delta\rho_{max} = 0.51$ e Å^{-3}
	$\Delta\rho_{min} = -0.68$ 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	$U_{iso}*/U_{eq}$
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 (Å^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)

Geometric parameters (Å, º)

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)

N2i—Co1—Cl1i	91.57 (6)	C6—C5—C4	121.3 (3)
N2—Co1—Cl1i	87.57 (6)	C4—C3—C2	116.8 (3)
N3—Co1—Cl1i	92.37 (6)	C4—C3—H3A	121.6
N3i—Co1—Cl1i	88.39 (6)	C2—C3—H3A	121.6
N2i—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
N3i—Co1—Cl1	92.37 (6)	C5—C6—C7	118.6 (3)
Cl1i—Co1—Cl1	178.70 (3)	C5—C6—H6A	120.7
C1—S1—C1i	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)	C1i—S1—C1—N1	177.9 (2)
N3i—Co1—N3—C5	170.5 (2)	C1i—S1—C1—N2	−3.15 (18)
Cl1i—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)
N3i—Co1—N3—C9	−9.30 (19)	Co1—N3—C5—C6	−177.6 (2)
Cl1i—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)
N2i—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)
Cl1i—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)
N2i—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)
Cl1i—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 code: (i) −x+1, y, −z+1/2.

Hydrogen-bond geometry (Å, º)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
C2—H2A⋯Cl1[ii]	0.93	2.63	3.546 (3)	170
C3—H3A⋯Cl1[iii]	0.93	2.73	3.584 (3)	154
C7—H7A⋯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.