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Crystal structure of bis[4-(4-chlorobenzyl)pyridine-κN]bis(thiocyanato-κN)zinc

Julia Werner,* Tristan Neumann, Inke Jess and Christian Näther

Institut für Anorganische Chemie, Christian-Albrechts-Universität Kiel, Max-Eyth-Strasse 2, 24118 Kiel, Germany. *Correspondence e-mail: jwerner@ac.uni-kiel.de

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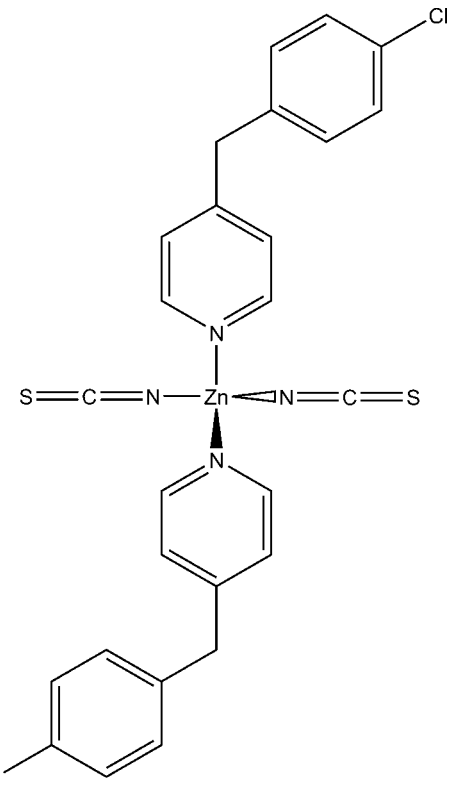
In the crystal structure of the title compound, $[Zn(NCS)_2(C_{12}H_{10}ClN)_2]$, the Zn^{2+} cation is *N*-coordinated by two terminally bonded thiocyante anions and by two 4-(4-chlorobenzyl)pyridine ligands within a slightly distorted tetrahedron. The asymmetric unit consists of half of the discrete complex, the central Zn^{2+} cation of which is located on a twofold rotation axis. The discrete complexes are linked into layers *via* a weak intermolecular hydrogen-bonding interaction, with a H···Cl distance of 2.85 Å and a C—H···Cl angle of 151°. These layers extend parallel to the *ab* plane and are held together by dispersion forces only.

Keywords: crystal structure; zinc complex; thiocyanate; tetrahedral coordination; hydrogen bonding.

CCDC reference: 1021253

1. Related literature

For related crystal structures with thiocyanate ligands and Zn^{2+} in a tetrahedral coordination sphere, see: Fettouhi *et al.* (2002); Kong *et al.* (2010); Zhu *et al.* (2008).



2. Experimental

2.1. Crystal data

$[Zn(NCS)_2(C_{12}H_{10}ClN)_2]$
M_r = 588.85
Monoclinic, $C2/c$
a = 29.094 (3) Å
b = 4.9911 (3) Å
c = 18.312 (2) Å
β = 98.867 (8)°
V = 2627.3 (4) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.32 mm^{-1}
T = 150 K
0.12 × 0.08 × 0.07 mm

2.2. Data collection

Stoe IPDS-2 diffractometer
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
T_{min} = 0.879, T_{max} = 0.906
8309 measured reflections
2570 independent reflections
1773 reflections with $I > 2\sigma(I)$
R_{int} = 0.077

2.3. Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.063
$wR(F^2)$ = 0.148
S = 1.08
2570 reflections
159 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.68 e Å^{-3}
$\Delta\rho_{min}$ = −0.46 e Å^{-3}

Data collection: *X-AREA* (Stoe, 2008); cell refinement: *X-AREA*; data reduction: *X-AREA*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *XP* in *SHELXTL* (Sheldrick, 2008) and *DIAMOND* (Brandenburg, 2011); software used to prepare material for publication: *publCIF* (Westrip, 2010).

Acknowledgements

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Supporting information for this paper is available from the IUCr electronic archives (Reference: WM5054).

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supporting information

Acta Cryst. (2014). E**70**, m355–m356 [doi:10.1107/S160053681402039X]

Crystal structure of bis[4-(4-chlorobenzyl)pyridine-κN]bis(thiocyanato-κN)zinc

Julia Werner, Tristan Neumann, Inke Jess and Christian Näther

S1. Synthesis and crystallization

$ZnSO_4 \cdot H_2O$ was purchased from Merck and 4-(4-chlorobenzyl)pyridine and $Ba(NCS)_2 \cdot 3H_2O$ were purchased from Alfa Aesar. $Zn(NCS)_2$ was synthesized by stirring 17.5 g (57.00 mmol) $Ba(NCS)_2 \cdot 3H_2O$ and 10.23 g (57.00 mmol) $ZnSO_4 \cdot H_2O$ in 300 mL water at RT for three hours. The white residue of $BaSO_4$ was filtered off, and the solution was evaporated by heating. The homogeneity of the product was investigated by X-ray powder diffraction and elemental analysis. The title compound was prepared by the reaction of (0.6 mmol) 108.9 mg $Zn(NCS)_2$ and (0.15 mmol) 105.5 μL 4-(4-chlorobenzyl)pyridine in 1.5 mL acetonitrile at RT. After few days, colorless needle-like crystals of the title compound were obtained.

S2. Refinement

Hydrogen atoms were positioned with idealized geometry and were refined with $U_{iso}(H) = 1.2\ U_{eq}(C)$ using a riding model with C—H = 0.95 Å for aromatic and C—H = 0.99 Å for methylene H atoms.

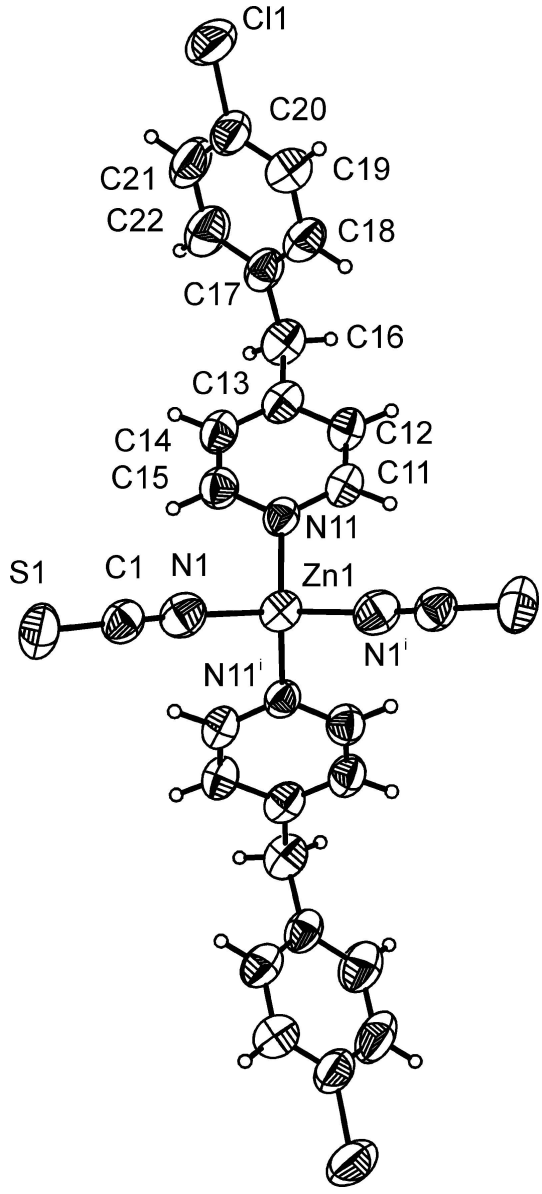

Figure 1

Molecular structure of the title compound with atom labelling and displacement ellipsoids drawn at the 50% probability level. [Symmetry code: i) -x, y, -z+1/2.]

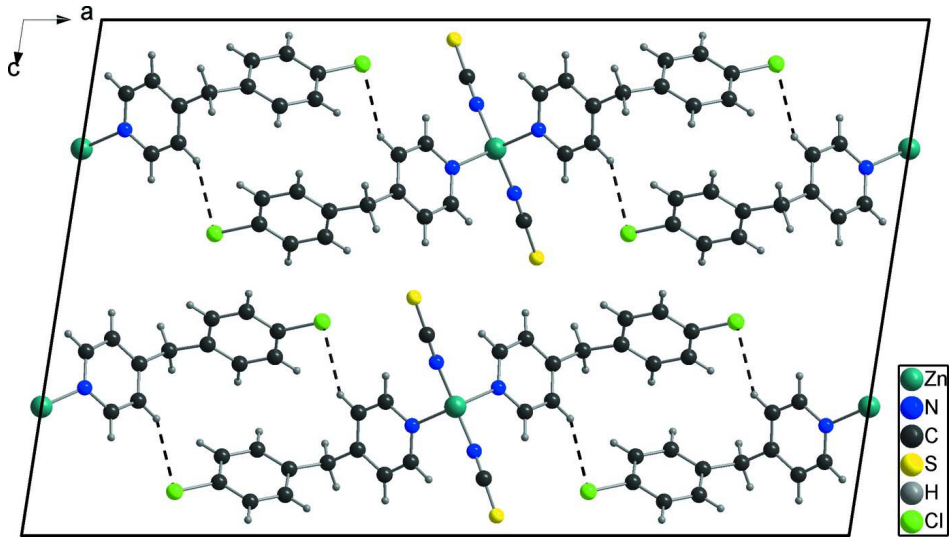

Figure 2

Crystal structure of the title compound in a projection along the *b* axis.

Bis[4-(4-chlorobenzyl)pyridine-*κN*]bis(thiocyanato-*κN*)zinc

Crystal data

$[Zn(NCS)_2(C_{12}H_{10}ClN)_2]$
$M_r = 588.85$
Monoclinic, *C*2/*c*
Hall symbol: -C 2yc
$a = 29.094$ (3) Å
$b = 4.9911$ (3) Å
$c = 18.312$ (2) Å
$\beta = 98.867$ (8)°
$V = 2627.3$ (4) Å^3
$Z = 4$
$F(000) = 1200$
$D_x = 1.489$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$\theta = 1.4$–26.0°
$\mu = 1.32$ mm^{-1}
$T = 150$ K
Needle, colourless
0.12 × 0.08 × 0.07 mm

Data collection

Stoe IPDS-2 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: numerical (*X-SHAPE* and *X-RED32*; Stoe, 2008)
$T_{min} = 0.879$, $T_{max} = 0.906$
8309 measured reflections
2570 independent reflections
1773 reflections with $I > 2\sigma(I)$
$R_{int} = 0.077$
$\theta_{max} = 26.0°$, $\theta_{min} = 1.4°$
$h = -35 \rightarrow 35$
$k = -6 \rightarrow 6$
$l = -22 \rightarrow 21$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.063$
$wR(F^2) = 0.148$
$S = 1.08$
2570 reflections
159 parameters
0 restraints
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
$w = 1/[\sigma^2(F_o^2) + (0.0577P)^2 + 3.849P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.68$ e Å^{-3}
$\Delta\rho_{min} = -0.46$ e Å^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2\text{sigma}(F^2)$ 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 (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Zn1	0.0000	−0.30960 (18)	0.2500	0.0536 (3)
N1	0.03121 (14)	−0.4936 (9)	0.3362 (3)	0.0632 (11)
C1	0.04787 (16)	−0.6043 (11)	0.3893 (3)	0.0564 (12)
S1	0.07036 (5)	−0.7701 (3)	0.46152 (9)	0.0767 (5)
N11	0.04855 (12)	−0.0763 (8)	0.2133 (2)	0.0482 (9)
C11	0.04345 (16)	0.0091 (10)	0.1431 (3)	0.0547 (12)
H11	0.0185	−0.0612	0.1088	0.066*
C12	0.07260 (16)	0.1938 (10)	0.1181 (3)	0.0552 (11)
H12	0.0671	0.2525	0.0682	0.066*
C13	0.11000 (16)	0.2929 (10)	0.1667 (3)	0.0551 (12)
C14	0.11577 (16)	0.2014 (12)	0.2384 (3)	0.0591 (13)
H14	0.1411	0.2640	0.2731	0.071*
C15	0.08515 (16)	0.0205 (10)	0.2597 (3)	0.0561 (12)
H15	0.0899	−0.0395	0.3096	0.067*
C16	0.14365 (18)	0.4920 (11)	0.1412 (3)	0.0654 (14)
H16A	0.1505	0.6354	0.1786	0.078*
H16B	0.1289	0.5759	0.0944	0.078*
C17	0.18905 (16)	0.3592 (11)	0.1292 (3)	0.0600 (14)
C18	0.18905 (17)	0.1565 (13)	0.0792 (3)	0.0688 (15)
H18	0.1603	0.0952	0.0529	0.083*
C19	0.22994 (19)	0.0380 (15)	0.0660 (4)	0.0815 (18)
H19	0.2292	−0.1021	0.0307	0.098*
C20	0.27132 (18)	0.1235 (16)	0.1039 (4)	0.0776 (18)
C21	0.2727 (2)	0.3210 (18)	0.1536 (4)	0.093 (2)
H21	0.3017	0.3783	0.1800	0.112*
C22	0.2313 (2)	0.4443 (15)	0.1669 (4)	0.0854 (19)
H22	0.2324	0.5859	0.2018	0.103*
Cl1	0.32321 (5)	−0.0187 (5)	0.08551 (11)	0.1091 (8)

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn1	0.0483 (4)	0.0500 (5)	0.0615 (5)	0.000	0.0050 (3)	0.000
N1	0.058 (2)	0.057 (3)	0.074 (3)	0.006 (2)	0.008 (2)	0.004 (2)
C1	0.051 (2)	0.055 (3)	0.061 (3)	−0.003 (2)	0.003 (2)	−0.007 (3)
S1	0.0709 (8)	0.0855 (12)	0.0657 (9)	−0.0042 (7)	−0.0148 (7)	0.0094 (8)

N11	0.0429 (18)	0.050 (2)	0.050 (2)	0.0061 (17)	0.0016 (16)	−0.0037 (19)
C11	0.048 (2)	0.053 (3)	0.059 (3)	0.001 (2)	−0.004 (2)	−0.002 (2)
C12	0.053 (2)	0.051 (3)	0.058 (3)	−0.001 (2)	−0.004 (2)	0.004 (3)
C13	0.051 (2)	0.041 (3)	0.071 (3)	0.005 (2)	0.001 (2)	−0.006 (3)
C14	0.052 (2)	0.063 (3)	0.059 (3)	−0.002 (2)	−0.004 (2)	−0.007 (3)
C15	0.055 (3)	0.058 (3)	0.053 (3)	0.001 (2)	0.004 (2)	−0.005 (2)
C16	0.064 (3)	0.048 (3)	0.081 (4)	−0.007 (2)	0.003 (3)	−0.002 (3)
C17	0.050 (3)	0.061 (4)	0.066 (3)	−0.011 (2)	−0.003 (2)	0.014 (3)
C18	0.049 (3)	0.076 (4)	0.078 (4)	−0.008 (3)	−0.001 (2)	0.001 (3)
C19	0.061 (3)	0.103 (5)	0.080 (4)	0.009 (3)	0.008 (3)	0.000 (4)
C20	0.050 (3)	0.110 (5)	0.069 (4)	0.004 (3)	−0.001 (3)	0.027 (4)
C21	0.051 (3)	0.132 (6)	0.089 (5)	−0.024 (4)	−0.012 (3)	0.034 (5)
C22	0.066 (3)	0.091 (5)	0.093 (5)	−0.017 (3)	−0.006 (3)	−0.003 (4)
Cl1	0.0565 (8)	0.168 (2)	0.1023 (13)	0.0255 (10)	0.0118 (8)	0.0551 (14)

Geometric parameters (Å, º)

Zn1—N1^{i}	1.928 (5)	C15—H15	0.9500
Zn1—N1	1.928 (5)	C16—C17	1.524 (7)
Zn1—N11	2.024 (4)	C16—H16A	0.9900
Zn1—N11^{i}	2.024 (4)	C16—H16B	0.9900
N1—C1	1.157 (6)	C17—C18	1.363 (8)
C1—S1	1.611 (6)	C17—C22	1.380 (7)
N11—C11	1.342 (6)	C18—C19	1.383 (8)
N11—C15	1.346 (6)	C18—H18	0.9500
C11—C12	1.377 (7)	C19—C20	1.363 (8)
C11—H11	0.9500	C19—H19	0.9500
C12—C13	1.386 (7)	C20—C21	1.338 (10)
C12—H12	0.9500	C20—Cl1	1.747 (6)
C13—C14	1.375 (7)	C21—C22	1.408 (10)
C13—C16	1.518 (7)	C21—H21	0.9500
C14—C15	1.367 (7)	C22—H22	0.9500
C14—H14	0.9500		
N1^{i}—Zn1—N1	123.1 (3)	C14—C15—H15	118.6
N1^{i}—Zn1—N11	105.49 (17)	C13—C16—C17	112.0 (4)
N1—Zn1—N11	106.33 (16)	C13—C16—H16A	109.2
N1^{i}—Zn1—N11^{i}	106.32 (16)	C17—C16—H16A	109.2
N1—Zn1—N11^{i}	105.49 (17)	C13—C16—H16B	109.2
N11—Zn1—N11^{i}	109.7 (2)	C17—C16—H16B	109.2
C1—N1—Zn1	176.6 (4)	H16A—C16—H16B	107.9
N1—C1—S1	177.6 (5)	C18—C17—C22	118.2 (5)
C11—N11—C15	116.9 (4)	C18—C17—C16	120.6 (4)
C11—N11—Zn1	121.4 (3)	C22—C17—C16	121.2 (6)
C15—N11—Zn1	121.4 (3)	C17—C18—C19	121.6 (5)
N11—C11—C12	123.2 (4)	C17—C18—H18	119.2
N11—C11—H11	118.4	C19—C18—H18	119.2
C12—C11—H11	118.4	C20—C19—C18	119.5 (7)

C11—C12—C13	119.3 (5)	C20—C19—H19	120.2
C11—C12—H12	120.4	C18—C19—H19	120.2
C13—C12—H12	120.4	C21—C20—C19	120.6 (6)
C14—C13—C12	117.5 (5)	C21—C20—Cl1	119.6 (5)
C14—C13—C16	121.5 (5)	C19—C20—Cl1	119.7 (6)
C12—C13—C16	121.0 (5)	C20—C21—C22	120.2 (6)
C15—C14—C13	120.3 (5)	C20—C21—H21	119.9
C15—C14—H14	119.9	C22—C21—H21	119.9
C13—C14—H14	119.9	C17—C22—C21	119.9 (7)
N11—C15—C14	122.9 (5)	C17—C22—H22	120.1
N11—C15—H15	118.6	C21—C22—H22	120.1

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