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Dichloridobis(2-methyl-1,3-benzothiazole-κN)zinc(II)

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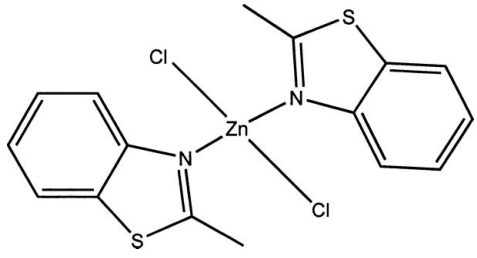
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.004 Å; R factor = 0.026; wR factor = 0.076; data-to-parameter ratio = 15.5.

The reaction of 4-[(2-mercaptophenyl)amino]pent-3-en-2-one with $ZnCl_2$ in CH_2Cl_2 gives the title complex, $[ZnCl_2(C_8H_7NS)_2]$, as a brown crystalline product in which 4-[(2-mercaptophenyl)amino]pent-3-en-2-one has been converted into 2-methylbenzothiazole. The Zn atom is coordinated by two chloride ions and binds to the N atoms of two 2-methylbenzothiazole ligands in a distorted tetrahedral arrangement.

Related literature

The synthesis of the complex is described by Peyronel & Giusti (1981). For other benzothiazole derivatives, see: Duff *et al.* (1969); Alyea & Malek (1985); Dey *et al.* (1995); Mueller *et al.* (1996).



Experimental

Crystal data

$[ZnCl_2(C_8H_7NS)_2]$
M_r = 434.72
Triclinic, $P\overline{1}$
a = 8.4063 (4) Å
b = 9.0758 (7) Å
c = 12.8451 (6) Å
α = 72.820 (4)°
β = 78.778 (4)°
γ = 69.938 (5)°
V = 874.70 (9) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 1.95 mm^{-1}
T = 293 (2) K
0.45 × 0.40 × 0.35 mm

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer
Absorption correction: ψ scan (*ABSCALC*; McArdle & Daly, 1999)
T_{min} = 0.433, T_{max} = 0.506
3567 measured reflections
3236 independent reflections
2798 reflections with $I > 2\sigma(I)$
R_{int} = 0.009
3 standard reflections
frequency: 60 min
intensity decay: <2%

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.026
$wR(F^2)$ = 0.076
S = 1.07
3236 reflections
209 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.44 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989); cell refinement: *CAD-4 Software*; data reduction: *XCAD* (McArdle, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: SJ2323).

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supplementary materials

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Dichloridobis(2-methyl-1,3-benzothiazole-*κN*)zinc(II)

S.-G. Roh and J. H. Jeong

Comment

Benzothiazole derivatives and their complexes (Duff *et al.*, 1969; Peyronel & Giusti, 1981; Alyea & Malek, 1985; Dey *et al.*, 1995; Mueller *et al.*, 1996) have been reported but the structure of the title compound has not been reported previously. The benzothiazoline derivatives were formed through the cyclocondensation of 2,4-pentanedione with 2-aminobenzenethiol by refluxing in EtOH or DMSO (Alyea & Malek, 1985; Dey *et al.*, 1995). However, the title complex was obtained here by the reaction of the ligand formed by the cyclocondensation of 4-(2-mercaptophenyl)amino-3-penten-2-one with $ZnCl_2$. The geometry around the zinc atom is a distorted tetrahedron containing two nitrogen atoms from 2-methylbenzothiazole ligands and two chloride ions. The planes of the benzothiazoles are almost perpendicular to each other [(83.71 (8)].

Experimental

To a solution of $ZnCl_2$ (0.08 g, 0.59 mmol) in CH_2Cl_2 (25 ml) was added dropwise a solution of 4-(2-mercaptophenyl)amino-3-penten-2-one (0.12 g, 1.2 mmol) in CH_2Cl_2 (25 ml). The mixture was stirred for 48 h at room temperature. The brown solution was filtered off and dried *in vacuo*. The residue was washed with ether to give a brown solid. Diffusion of n-hexane into a brown solution in CH_2Cl_2 gave brown crystals within two weeks. Yield; 0.13 g (51%). Anal. Calcd. for $C_{16}H_{14}Cl_2N_2S_2Zn$: C, 44.21; H, 3.25; N, 6.44; S, 14.75. Found: C, 44.01; H, 3.27; N, 6.52; S, 14.74. ^{1}H NMR ($CDCl_3$); 7.48(d, 1H, 2-benz, J = 8.1 Hz), 7.42(d, 1H, 7-benz, J = 7.8 Hz), 7.01(m, 1H, 6-benz), 6.92(m, 1H, 5-benz), 2.21(s, 3H, CH_3).

Refinement

All H-atoms were positioned geometrically and refined using a riding model with d(C—H) = 0.93 Å, U_{iso} = 1.2U_{eq}(C) for aromatic and 0.96 Å, U_{iso} = 1.5U_{eq}(C) for CH_3 atoms.

Figures

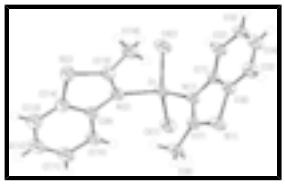


Fig. 1. A view of Dichlorobis(2-methylbenzothiazole-κ*N*)zinc(II). Displacement ellipsoids are drawn at the 40% probability level.

Dichloridobis(2-methyl-1,3-benzothiazole-κ*N*)zinc(II)

Crystal data

$[ZnCl_2(C_8H_7NS)_2]$ $Z = 2$

M_r = 434.72	F_{000} = 440
Triclinic, $P\bar{1}$	D_x = 1.651 Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.4063 (4) Å	Cell parameters from 25 reflections
b = 9.0758 (7) Å	θ = 9.6–13.1º
c = 12.8451 (6) Å	μ = 1.95 mm^{-1}
α = 72.820 (4)º	T = 293 (2) K
β = 78.778 (4)º	Block, light brown
γ = 69.938 (5)º	0.45 × 0.40 × 0.35 mm
V = 874.70 (9) Å^3	

Data collection

Enraf–Nonius CAD-4 four-circle diffractometer	2798 reflections with $I > 2\sigma(I)$
Radiation source: fine-focus sealed tube	R_{int} = 0.009
Monochromator: graphite	θ_{max} = 25.5º
T = 293(2) K	θ_{min} = 1.7º
scintillation counter scans	h = −10→9
Absorption correction: ψ scan (ABSCALC; McArdle & Daly, 1999)	k = −10→0
T_{min} = 0.433, T_{max} = 0.506	l = −15→14
3567 measured reflections	3 standard reflections
3236 independent reflections	every 60 min

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.026	H-atom parameters constrained
$wR(F^2)$ = 0.076	$w = 1/[\sigma^2(F_o^2) + (0.0463P)^2 + 0.2584P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.07	$(\Delta/\sigma)_{max}$ = 0.001
3236 reflections	$\Delta\rho_{max}$ = 0.44 e Å^{-3}
209 parameters	$\Delta\rho_{min}$ = −0.37 e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
Zn	0.27503 (3)	0.27750 (3)	0.228264 (19)	0.03207 (10)
Cl1	0.15138 (7)	0.51803 (6)	0.12194 (5)	0.04421 (15)
Cl2	0.52890 (7)	0.26472 (8)	0.26501 (5)	0.04639 (16)
N1	0.2735 (2)	0.0927 (2)	0.16511 (14)	0.0304 (4)

C1	0.4131 (3)	−0.0405 (2)	0.14713 (17)	0.0307 (4)
C2	0.5815 (3)	−0.0633 (3)	0.15698 (19)	0.0380 (5)
H2	0.6124	0.0123	0.1776	0.046*
C3	0.7029 (3)	−0.2024 (3)	0.1351 (2)	0.0430 (5)
H3	0.8169	−0.2198	0.1409	0.052*
C4	0.6574 (3)	−0.3154 (3)	0.1050 (2)	0.0472 (6)
H4	0.7415	−0.4079	0.0917	0.057*
C5	0.4921 (3)	−0.2947 (3)	0.0942 (2)	0.0463 (6)
H5	0.4627	−0.3708	0.0732	0.056*
C6	0.3691 (3)	−0.1555 (3)	0.11590 (18)	0.0347 (5)
S1	0.15172 (7)	−0.09114 (7)	0.10699 (5)	0.04242 (15)
C7	0.1320 (3)	0.0818 (2)	0.14470 (17)	0.0329 (4)
C8	−0.0381 (3)	0.2062 (3)	0.1512 (2)	0.0433 (5)
H8A	−0.0530	0.2452	0.2154	0.065*
H8B	−0.1257	0.1586	0.1553	0.065*
H8C	−0.0449	0.2947	0.0872	0.065*
N2	0.1375 (2)	0.2211 (2)	0.38233 (15)	0.0345 (4)
C9	−0.0114 (3)	0.3256 (3)	0.42531 (18)	0.0356 (5)
C10	−0.1195 (3)	0.4644 (3)	0.3654 (2)	0.0479 (6)
H10	−0.0964	0.4991	0.2900	0.058*
C11	−0.2629 (3)	0.5499 (4)	0.4209 (2)	0.0544 (7)
H11	−0.3380	0.6422	0.3817	0.065*
C12	−0.2970 (3)	0.5017 (4)	0.5329 (2)	0.0545 (7)
H12	−0.3931	0.5631	0.5680	0.065*
C13	−0.1922 (4)	0.3656 (4)	0.5930 (2)	0.0558 (7)
H13	−0.2153	0.3338	0.6687	0.067*
C14	−0.0490 (3)	0.2747 (3)	0.53832 (19)	0.0429 (5)
S2	0.10126 (9)	0.09379 (9)	0.58910 (5)	0.05466 (19)
C15	0.2041 (3)	0.0948 (3)	0.45884 (18)	0.0375 (5)
C16	0.3534 (3)	−0.0466 (3)	0.4400 (2)	0.0475 (6)
H16A	0.3268	−0.0968	0.3923	0.071*
H16B	0.3790	−0.1237	0.5088	0.071*
H16C	0.4503	−0.0100	0.4069	0.071*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Zn	0.03075 (15)	0.02626 (15)	0.03583 (16)	−0.00427 (10)	−0.00148 (10)	−0.00924 (10)
Cl1	0.0497 (3)	0.0269 (3)	0.0462 (3)	−0.0041 (2)	−0.0038 (2)	−0.0043 (2)
Cl2	0.0338 (3)	0.0505 (3)	0.0596 (4)	−0.0116 (2)	−0.0023 (2)	−0.0239 (3)
N1	0.0285 (8)	0.0249 (8)	0.0336 (9)	−0.0036 (7)	−0.0020 (7)	−0.0075 (7)
C1	0.0320 (10)	0.0241 (9)	0.0309 (10)	−0.0030 (8)	−0.0017 (8)	−0.0070 (8)
C2	0.0336 (11)	0.0340 (11)	0.0441 (12)	−0.0041 (9)	−0.0041 (9)	−0.0136 (10)
C3	0.0319 (11)	0.0382 (12)	0.0479 (13)	0.0021 (9)	−0.0019 (9)	−0.0111 (10)
C4	0.0469 (13)	0.0314 (11)	0.0504 (14)	0.0051 (10)	−0.0005 (11)	−0.0144 (10)
C5	0.0538 (14)	0.0303 (11)	0.0521 (15)	−0.0038 (10)	−0.0046 (11)	−0.0178 (10)
C6	0.0384 (11)	0.0270 (10)	0.0353 (11)	−0.0065 (8)	−0.0034 (9)	−0.0070 (8)
S1	0.0403 (3)	0.0349 (3)	0.0560 (4)	−0.0110 (2)	−0.0099 (3)	−0.0144 (3)

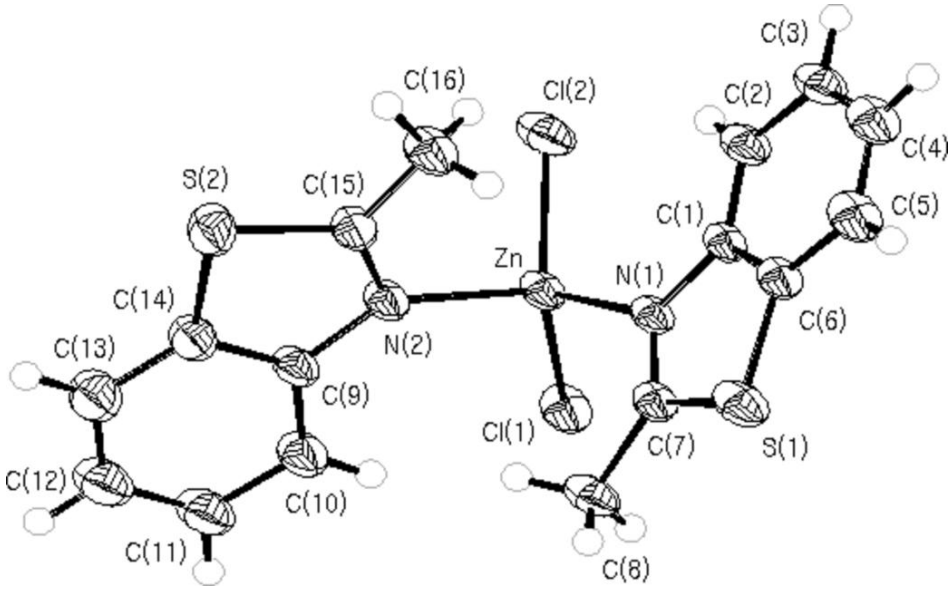
C7	0.0344 (11)	0.0281 (10)	0.0328 (11)	−0.0079 (8)	−0.0024 (8)	−0.0052 (8)
C8	0.0309 (11)	0.0375 (12)	0.0556 (14)	−0.0033 (9)	−0.0070 (10)	−0.0099 (11)
N2	0.0345 (9)	0.0321 (9)	0.0340 (9)	−0.0076 (7)	−0.0003 (7)	−0.0089 (7)
C9	0.0326 (10)	0.0393 (12)	0.0362 (11)	−0.0113 (9)	0.0010 (8)	−0.0135 (9)
C10	0.0422 (13)	0.0480 (14)	0.0418 (13)	−0.0029 (11)	0.0028 (10)	−0.0114 (11)
C11	0.0416 (13)	0.0555 (16)	0.0582 (17)	−0.0012 (12)	0.0001 (11)	−0.0219 (13)
C12	0.0441 (14)	0.0626 (17)	0.0605 (17)	−0.0120 (12)	0.0102 (12)	−0.0358 (14)
C13	0.0598 (16)	0.0687 (18)	0.0419 (14)	−0.0254 (14)	0.0141 (12)	−0.0238 (13)
C14	0.0459 (13)	0.0498 (14)	0.0356 (12)	−0.0174 (11)	0.0012 (10)	−0.0144 (10)
S2	0.0637 (4)	0.0547 (4)	0.0338 (3)	−0.0135 (3)	−0.0018 (3)	−0.0013 (3)
C15	0.0418 (12)	0.0370 (11)	0.0338 (11)	−0.0136 (9)	−0.0053 (9)	−0.0062 (9)
C16	0.0481 (14)	0.0349 (12)	0.0513 (14)	−0.0032 (10)	−0.0120 (11)	−0.0051 (10)

Geometric parameters (Å, °)

Zn—N1	2.0725 (17)	C8—H8B	0.9600
Zn—N2	2.1054 (18)	C8—H8C	0.9600
Zn—Cl1	2.2239 (6)	N2—C15	1.304 (3)
Zn—Cl2	2.2317 (6)	N2—C9	1.413 (3)
N1—C7	1.307 (3)	C9—C10	1.385 (3)
N1—C1	1.406 (2)	C9—C14	1.395 (3)
C1—C2	1.385 (3)	C10—C11	1.383 (3)
C1—C6	1.398 (3)	C10—H10	0.9300
C2—C3	1.389 (3)	C11—C12	1.378 (4)
C2—H2	0.9300	C11—H11	0.9300
C3—C4	1.381 (4)	C12—C13	1.361 (4)
C3—H3	0.9300	C12—H12	0.9300
C4—C5	1.366 (4)	C13—C14	1.398 (4)
C4—H4	0.9300	C13—H13	0.9300
C5—C6	1.393 (3)	C14—S2	1.727 (3)
C5—H5	0.9300	S2—C15	1.726 (2)
C6—S1	1.733 (2)	C15—C16	1.497 (3)
S1—C7	1.722 (2)	C16—H16A	0.9600
C7—C8	1.493 (3)	C16—H16B	0.9600
C8—H8A	0.9600	C16—H16C	0.9600
N1—Zn—N2	99.81 (7)	C7—C8—H8C	109.5
N1—Zn—Cl1	110.72 (5)	H8A—C8—H8C	109.5
N2—Zn—Cl1	114.11 (5)	H8B—C8—H8C	109.5
N1—Zn—Cl2	115.46 (5)	C15—N2—C9	111.04 (19)
N2—Zn—Cl2	105.13 (5)	C15—N2—Zn	121.06 (15)
Cl1—Zn—Cl2	111.12 (3)	C9—N2—Zn	126.32 (14)
C7—N1—C1	111.33 (18)	C10—C9—C14	120.0 (2)
C7—N1—Zn	121.60 (14)	C10—C9—N2	126.0 (2)
C1—N1—Zn	126.88 (14)	C14—C9—N2	113.9 (2)
C2—C1—C6	120.16 (19)	C11—C10—C9	118.2 (2)
C2—C1—N1	126.06 (19)	C11—C10—H10	120.9
C6—C1—N1	113.77 (18)	C9—C10—H10	120.9
C1—C2—C3	118.0 (2)	C12—C11—C10	121.5 (3)
C1—C2—H2	121.0	C12—C11—H11	119.3

C3—C2—H2	121.0	C10—C11—H11	119.3
C4—C3—C2	121.1 (2)	C13—C12—C11	121.1 (2)
C4—C3—H3	119.4	C13—C12—H12	119.5
C2—C3—H3	119.4	C11—C12—H12	119.5
C5—C4—C3	121.7 (2)	C12—C13—C14	118.4 (2)
C5—C4—H4	119.1	C12—C13—H13	120.8
C3—C4—H4	119.1	C14—C13—H13	120.8
C4—C5—C6	117.6 (2)	C9—C14—C13	120.7 (2)
C4—C5—H5	121.2	C9—C14—S2	109.57 (17)
C6—C5—H5	121.2	C13—C14—S2	129.7 (2)
C5—C6—C1	121.3 (2)	C15—S2—C14	90.28 (11)
C5—C6—S1	129.17 (19)	N2—C15—C16	125.0 (2)
C1—C6—S1	109.50 (15)	N2—C15—S2	115.14 (17)
C7—S1—C6	90.15 (10)	C16—C15—S2	119.83 (17)
N1—C7—C8	125.0 (2)	C15—C16—H16A	109.5
N1—C7—S1	115.20 (15)	C15—C16—H16B	109.5
C8—C7—S1	119.79 (16)	H16A—C16—H16B	109.5
C7—C8—H8A	109.5	C15—C16—H16C	109.5
C7—C8—H8B	109.5	H16A—C16—H16C	109.5
H8A—C8—H8B	109.5	H16B—C16—H16C	109.5
N2—Zn—N1—C7	64.80 (17)	N1—Zn—N2—C15	71.18 (18)
Cl1—Zn—N1—C7	−55.77 (17)	Cl1—Zn—N2—C15	−170.73 (15)
Cl2—Zn—N1—C7	176.88 (14)	Cl2—Zn—N2—C15	−48.73 (18)
N2—Zn—N1—C1	−109.69 (17)	N1—Zn—N2—C9	−124.47 (17)
Cl1—Zn—N1—C1	129.73 (16)	Cl1—Zn—N2—C9	−6.39 (19)
Cl2—Zn—N1—C1	2.38 (18)	Cl2—Zn—N2—C9	115.62 (17)
C7—N1—C1—C2	177.3 (2)	C15—N2—C9—C10	−175.2 (2)
Zn—N1—C1—C2	−7.7 (3)	Zn—N2—C9—C10	19.1 (3)
C7—N1—C1—C6	−2.2 (3)	C15—N2—C9—C14	2.4 (3)
Zn—N1—C1—C6	172.79 (14)	Zn—N2—C9—C14	−163.21 (16)
C6—C1—C2—C3	−0.1 (3)	C14—C9—C10—C11	0.6 (4)
N1—C1—C2—C3	−179.6 (2)	N2—C9—C10—C11	178.1 (2)
C1—C2—C3—C4	−0.3 (4)	C9—C10—C11—C12	1.3 (4)
C2—C3—C4—C5	0.7 (4)	C10—C11—C12—C13	−1.4 (5)
C3—C4—C5—C6	−0.7 (4)	C11—C12—C13—C14	−0.4 (4)
C4—C5—C6—C1	0.3 (4)	C10—C9—C14—C13	−2.3 (4)
C4—C5—C6—S1	178.36 (19)	N2—C9—C14—C13	179.8 (2)
C2—C1—C6—C5	0.1 (3)	C10—C9—C14—S2	176.8 (2)
N1—C1—C6—C5	179.7 (2)	N2—C9—C14—S2	−1.0 (3)
C2—C1—C6—S1	−178.33 (17)	C12—C13—C14—C9	2.2 (4)
N1—C1—C6—S1	1.2 (2)	C12—C13—C14—S2	−176.7 (2)
C5—C6—S1—C7	−178.3 (2)	C9—C14—S2—C15	−0.39 (18)
C1—C6—S1—C7	−0.02 (16)	C13—C14—S2—C15	178.6 (3)
C1—N1—C7—C8	−177.4 (2)	C9—N2—C15—C16	174.5 (2)
Zn—N1—C7—C8	7.3 (3)	Zn—N2—C15—C16	−19.0 (3)
C1—N1—C7—S1	2.2 (2)	C9—N2—C15—S2	−2.8 (2)
Zn—N1—C7—S1	−173.10 (9)	Zn—N2—C15—S2	163.76 (11)
C6—S1—C7—N1	−1.28 (17)	C14—S2—C15—N2	1.89 (19)
C6—S1—C7—C8	178.31 (19)	C14—S2—C15—C16	−175.5 (2)

Fig. 1


C(3)
C(16)
Cl(2)
C(2)
C(4)
S(2)
C(15)
C(1)
C(5)
Zn
N(1)
C(14)
C(6)
C(13)
N(2)
C(9)
Cl(1)
C(7)
S(1)
C(12)
C(10)
C(11)
C(8)