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Di-μ-thiocyanato-bis({2,4-dichloro-6-[2-(diethylamino)ethyliminomethyl]-phenolato}copper(II))

Xian-Wen Li[a]* and Yang Qiu[b]

[a]Department of Chemistry and Chemical Engineering, Minjiang University, Fuzhou 350108, People's Republic of China, and [b]Department of Chemistry and Physics, University of Science and Technology of China, Hefei 230026, People's Republic of China
Correspondence e-mail: xianwenfz@163.com

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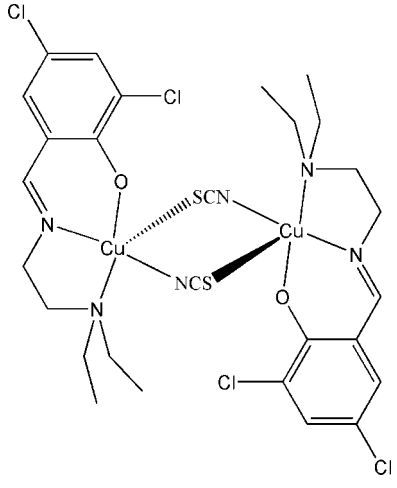
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.005 Å; R factor = 0.044; wR factor = 0.118; data-to-parameter ratio = 17.5.

The title compound, $[Cu_2(NCS)_2(C_{13}H_{17}Cl_2N_2O)_2]$, was obtained by the reaction of 3,5-dichlorosalicylaldehyde, *N*,*N*-diethylethane-1,2-diamine, sodium thiocyanate, and copper(II) acetate in an ethanol solution. It crystallizes as a centrosymmetric dimer with a very long Cu···S axial bond [2.972 (3) Å]. The Cu atom is five-coordinated by the three donor atoms of the Schiff base ligand, 2,4-dichloro-6-[(2-diethylaminoethylimino)methyl]phenol, one N atom of a thiocyanate group, and one S atom of a symmetry-related thiocyanate group, forming a slightly distorted square-pyramidal geometry.

Related literature

For the biological activity of Schiff base compounds, see: Panneerselvam *et al.* (2005); Shi *et al.* (2007); Singh *et al.* (2006, 2007); Zhong *et al.* (2006). For related literature, see: Allen *et al.* (1987).



Experimental

Crystal data

$[Cu_2(NCS)_2(C_{13}H_{17}Cl_2N_2O)_2]$
M_r = 409.81
Monoclinic, $P2_1/c$
a = 8.632 (2) Å
b = 14.115 (3) Å
c = 14.002 (3) Å
β = 90.491 (4)°
V = 1706.0 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.72 mm^{-1}
T = 293 (2) K
0.20 × 0.17 × 0.16 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.725, T_{max} = 0.771
13451 measured reflections
3516 independent reflections
3046 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.044
$wR(F^2)$ = 0.118
S = 1.06
3516 reflections
201 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.34 e Å^{-3}
$\Delta\rho_{min}$ = −0.67 e Å^{-3}

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; 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: SU2031).

References

Allen, F. H., Kennard, O., Watson, D. G., Brammer, L., Orpen, A. G. & Taylor, R. (1987). *J. Chem. Soc. Perkin Trans. 2*, pp. S1–19.
Bruker (2001). *SMART* and *SAINT*. Bruker AXS Inc., Madison, Wisconsin, USA.
Panneerselvam, P., Nair, R. R., Vijayalakshmi, G., Subramanian, E. H. & Krishnan, S. (2005). *Eur. J. Med. Chem.* **40**, 225–229.
Sheldrick, G. M. (2001). *SHELXTL*. Version 5.0. Bruker AXS Inc., Madison, Wisconsin, USA.
Sheldrick, G. M. (2004). *SADABS*. University of Göttingen, Germany.
Shi, L., Ge, H.-M., Tan, S.-H., Li, H.-Q., Song, Y.-C., Zhu, H.-L. & Tan, R.-X. (2007). *Eur. J. Med. Chem.* **42**, 558–564.
Singh, K., Barwa, M. S. & Tyagi, P. (2006). *Eur. J. Med. Chem.* **41**, 147–153.
Singh, K., Barwa, M. S. & Tyagi, P. (2007). *Eur. J. Med. Chem.* **42**, 394–402.
Zhong, X., Yi, J., Sun, J., Wei, H.-L., Liu, W.-S. & Yu, K.-B. (2006). *Eur. J. Med. Chem.* **41**, 1090–1092.

supplementary materials

Acta Cryst. (2008). E**64**, m218 [**doi:10.1107/S1600536807063325**]

Di-μ-thiocyanato-bis({2,4-dichloro-6-[2-(diethylamino)ethyliminomethyl]phenolato}copper(II))

X.-W. Li and Y. Qiu

Comment

Schiff base compounds have been reported to have excellent biological activity (Shi *et al.*, 2007; Panneerselvam *et al.*, 2005). Metal complexes derived from the Schiff bases have also been shown to have excellent biological activity (Singh *et al.*, 2006, 2007; Zhong *et al.*, 2006). As part of our investigations of the structures of metal complexes derived from Schiff bases, we report herein the crystal structure of the title copper complex, (I).

Compound (I) is a centrosymmetric dinuclear copper(II) complex (Fig. 1). The Cu atom is five-coordinated by the three donor atoms (O1, N1 and N2) of the Schiff base ligand 2,4-dichloro-6-[(2-diethylaminoethylimino)methyl]phenol, one N atom of a thiocyanate group, and one S atom of the centrosymmetrically related thiocyanate group, so forming a slightly distorted square-pyramidal geometry. The Cu atom is displaced out of the best least-squares plane defined by the four basal donor atoms by 0.123 (2) Å. Apart from the long Cu···S axial bond [2.972 (3) Å], the other coordination bond distances and angles are within normal ranges (Allen *et al.*, 1987).

Experimental

The title compound was obtained by the reaction of equimolar amounts of 3,5-dichlorosalicylaldehyde, *N*,*N*-diethylethane-1,2-diamine, sodium thiocyanate, and copper acetate in an ethanol solution. Blue block-like single crystals were obtained by slow evaporation of the filtrate in air.

Refinement

H atoms were positioned geometrically and treated as riding atoms, with C—H = 0.93–0.97Å and U_{iso}(H) = 1.2 (1.5 for methyl groups) times U_{eq}(C).

Figures

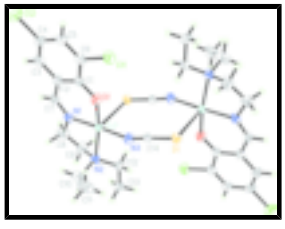


Fig. 1. The molecular structure of complex (I), with atom labels and 30% probability displacement ellipsoids for non-H atoms.

Di-μ-thiocyanato-bis({2,4-dichloro-6-[2- (diethylamino)ethyliminomethyl]phenolato}copper(II))

Crystal data

$[Cu_2(NCS)_2(C_{13}H_{17}Cl_2N_2O)_2]$	F_{000} = 836
M_r = 409.81	D_x = 1.596 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å
a = 8.632 (2) Å	Cell parameters from 6502 reflections
b = 14.115 (3) Å	θ = 2.4–27.7º
c = 14.002 (3) Å	μ = 1.72 mm^{-1}
β = 90.491 (4)º	T = 293 (2) K
V = 1706.0 (6) Å^3	Block, blue
Z = 4	0.20 × 0.17 × 0.16 mm

Data collection

Bruker SMART 1K CCD area-detector diffractometer	3516 independent reflections
Radiation source: fine-focus sealed tube	3046 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 293(2) K	θ_{max} = 26.5º
ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −10→10
T_{min} = 0.725, T_{max} = 0.771	k = −17→17
13451 measured reflections	l = −17→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.044	H-atom parameters constrained
$wR(F^2)$ = 0.118	$w = 1/[\sigma^2(F_o^2) + (0.058P)^2 + 2.2599P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.06	$(\Delta/\sigma)_{max}$ = 0.001
3516 reflections	$\Delta\rho_{max}$ = 1.34 e Å^{-3}
201 parameters	$\Delta\rho_{min}$ = −0.67 e Å^{-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 (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.13607 (4)	0.16622 (3)	0.06969 (3)	0.03794 (14)
O1	−0.0063 (3)	0.22330 (17)	−0.01736 (16)	0.0489 (6)
N1	0.1165 (3)	0.2683 (2)	0.16144 (18)	0.0410 (6)
N2	0.3213 (3)	0.1254 (2)	0.1558 (2)	0.0444 (6)
N3	0.1658 (4)	0.0673 (2)	−0.0253 (2)	0.0477 (7)
Cl1	−0.21684 (12)	0.25400 (7)	−0.17482 (6)	0.0585 (3)
Cl2	−0.33491 (14)	0.58481 (8)	−0.00046 (9)	0.0716 (3)
S1	0.10771 (10)	−0.05935 (7)	−0.17261 (6)	0.0476 (2)
C1	−0.0792 (4)	0.3018 (2)	−0.0076 (2)	0.0396 (7)
C2	−0.0634 (4)	0.3643 (2)	0.0713 (2)	0.0419 (7)
C3	−0.1427 (4)	0.4516 (3)	0.0723 (3)	0.0498 (8)
H3	−0.1293	0.4927	0.1236	0.060*
C4	−0.2389 (4)	0.4760 (3)	−0.0013 (3)	0.0510 (9)
C5	−0.2616 (4)	0.4157 (3)	−0.0785 (3)	0.0490 (8)
H5	−0.3279	0.4327	−0.1283	0.059*
C6	−0.1850 (4)	0.3311 (2)	−0.0802 (2)	0.0435 (7)
C7	0.0343 (4)	0.3430 (2)	0.1518 (2)	0.0450 (8)
H7	0.0382	0.3872	0.2010	0.054*
C8	0.2109 (5)	0.2585 (3)	0.2480 (3)	0.0621 (11)
H8A	0.2939	0.3048	0.2477	0.075*
H8B	0.1477	0.2698	0.3038	0.075*
C9	0.2742 (7)	0.1654 (3)	0.2521 (3)	0.0741 (14)
H9A	0.1983	0.1234	0.2803	0.089*
H9B	0.3644	0.1662	0.2939	0.089*
C10	0.4629 (6)	0.1738 (4)	0.1273 (4)	0.0883 (17)
H10A	0.4497	0.2409	0.1395	0.106*
H10B	0.5466	0.1518	0.1683	0.106*
C11	0.5099 (6)	0.1626 (5)	0.0307 (4)	0.114 (3)
H11A	0.5219	0.0964	0.0168	0.171*
H11B	0.6069	0.1945	0.0214	0.171*
H11C	0.4328	0.1893	−0.0111	0.171*
C12	0.3232 (5)	0.0217 (3)	0.1662 (3)	0.0581 (10)
H12A	0.3370	−0.0057	0.1034	0.070*
H12B	0.2221	0.0020	0.1886	0.070*
C13	0.4448 (5)	−0.0206 (3)	0.2326 (3)	0.0661 (11)
H13A	0.5456	0.0009	0.2140	0.099*
H13B	0.4406	−0.0884	0.2288	0.099*
H13C	0.4249	−0.0010	0.2970	0.099*

C14	0.1388 (3)	0.0159 (2)	−0.0861 (2)	0.0361 (6)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0441 (2)	0.0384 (2)	0.0312 (2)	−0.00149 (16)	−0.00774 (16)	−0.00402 (15)
O1	0.0670 (16)	0.0426 (13)	0.0368 (12)	0.0082 (11)	−0.0161 (11)	−0.0057 (10)
N1	0.0423 (15)	0.0514 (16)	0.0293 (13)	−0.0062 (12)	−0.0044 (11)	−0.0049 (11)
N2	0.0438 (15)	0.0452 (16)	0.0441 (15)	−0.0062 (12)	−0.0110 (12)	0.0016 (12)
N3	0.0522 (17)	0.0497 (17)	0.0410 (15)	0.0019 (13)	−0.0060 (13)	−0.0096 (13)
Cl1	0.0706 (6)	0.0653 (6)	0.0392 (5)	0.0004 (5)	−0.0145 (4)	0.0011 (4)
Cl2	0.0714 (7)	0.0571 (6)	0.0867 (8)	0.0217 (5)	0.0217 (6)	0.0136 (5)
S1	0.0468 (5)	0.0543 (5)	0.0417 (5)	−0.0034 (4)	0.0008 (4)	−0.0165 (4)
C1	0.0425 (17)	0.0404 (17)	0.0358 (16)	−0.0053 (14)	0.0011 (13)	0.0028 (13)
C2	0.0423 (17)	0.0428 (17)	0.0406 (17)	−0.0016 (14)	0.0037 (13)	0.0003 (14)
C3	0.052 (2)	0.048 (2)	0.050 (2)	0.0001 (16)	0.0146 (16)	−0.0041 (16)
C4	0.0471 (19)	0.047 (2)	0.059 (2)	0.0088 (15)	0.0167 (17)	0.0094 (17)
C5	0.0420 (18)	0.056 (2)	0.049 (2)	0.0045 (15)	0.0060 (15)	0.0153 (16)
C6	0.0444 (17)	0.050 (2)	0.0359 (16)	−0.0046 (14)	0.0023 (13)	0.0064 (14)
C7	0.0479 (19)	0.049 (2)	0.0379 (17)	−0.0054 (15)	0.0026 (14)	−0.0117 (14)
C8	0.056 (2)	0.094 (3)	0.0361 (18)	0.006 (2)	−0.0135 (16)	−0.0129 (19)
C9	0.111 (4)	0.066 (3)	0.045 (2)	0.014 (2)	−0.031 (2)	−0.0106 (19)
C10	0.057 (3)	0.117 (5)	0.091 (4)	−0.022 (3)	−0.014 (3)	0.028 (3)
C11	0.057 (3)	0.187 (7)	0.100 (5)	−0.016 (4)	0.020 (3)	−0.061 (5)
C12	0.052 (2)	0.045 (2)	0.077 (3)	0.0055 (16)	−0.0224 (19)	−0.0060 (19)
C13	0.058 (2)	0.061 (2)	0.079 (3)	0.015 (2)	−0.020 (2)	0.000 (2)
C14	0.0315 (15)	0.0419 (17)	0.0350 (15)	0.0029 (12)	−0.0002 (12)	−0.0001 (13)

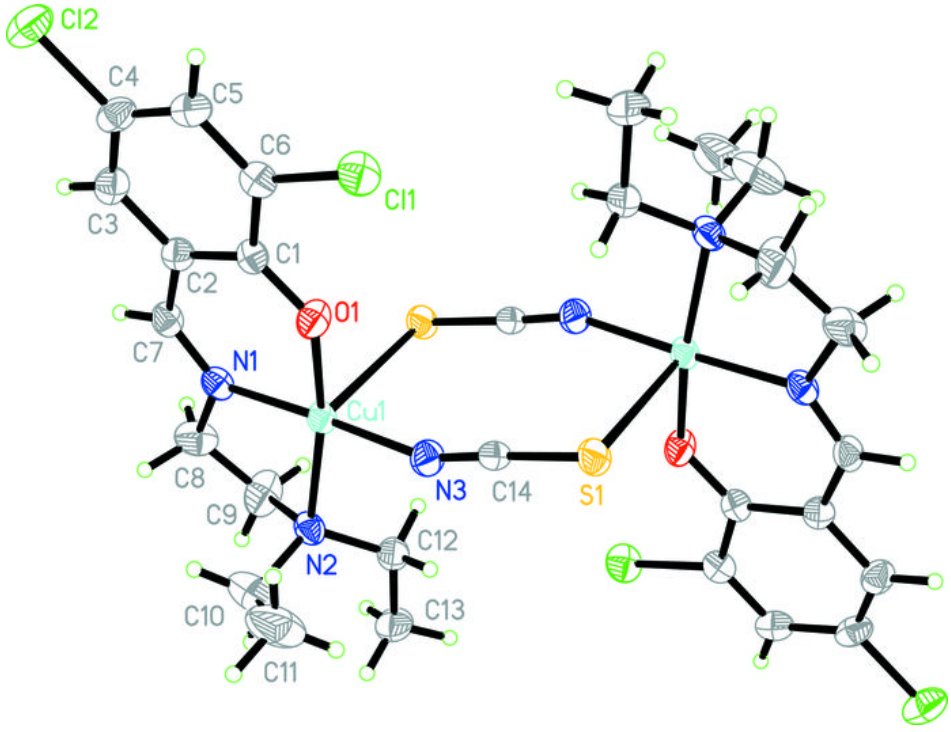
Geometric parameters (Å, °)

Cu1—O1	1.903 (2)	C4—C5	1.389 (6)
Cu1—N1	1.939 (3)	C5—C6	1.365 (5)
Cu1—N3	1.947 (3)	C5—H5	0.9300
Cu1—N2	2.076 (3)	C7—H7	0.9300
Cu1—S1^{i}	2.972 (3)	C8—C9	1.423 (6)
O1—C1	1.282 (4)	C8—H8A	0.9700
N1—C7	1.278 (4)	C8—H8B	0.9700
N1—C8	1.461 (4)	C9—H9A	0.9700
N2—C10	1.459 (5)	C9—H9B	0.9700
N2—C12	1.470 (5)	C10—C11	1.424 (8)
N2—C9	1.520 (5)	C10—H10A	0.9700
N3—C14	1.141 (4)	C10—H10B	0.9700
Cl1—C6	1.734 (4)	C11—H11A	0.9600
Cl2—C4	1.745 (4)	C11—H11B	0.9600
S1—C14	1.632 (3)	C11—H11C	0.9600
C1—C2	1.420 (5)	C12—C13	1.518 (5)
C1—C6	1.422 (5)	C12—H12A	0.9700
C2—C3	1.410 (5)	C12—H12B	0.9700

C2—C7	1.434 (5)	C13—H13A	0.9600
C3—C4	1.362 (5)	C13—H13B	0.9600
C3—H3	0.9300	C13—H13C	0.9600
O1—Cu1—N1	92.89 (11)	N1—C7—H7	117.3
O1—Cu1—N3	87.38 (11)	C2—C7—H7	117.3
N1—Cu1—N3	176.78 (12)	C9—C8—N1	109.4 (3)
O1—Cu1—N2	168.48 (11)	C9—C8—H8A	109.8
N1—Cu1—N2	83.82 (12)	N1—C8—H8A	109.8
N3—Cu1—N2	95.29 (12)	C9—C8—H8B	109.8
O1—Cu1—S1^{i}	93.98 (12)	N1—C8—H8B	109.8
N1—Cu1—S1^{i}	89.43 (12)	H8A—C8—H8B	108.2
N2—Cu1—S1^{i}	97.02 (12)	C8—C9—N2	114.4 (4)
N3—Cu1—S1^{i}	93.75 (12)	C8—C9—H9A	108.7
C1—O1—Cu1	127.8 (2)	N2—C9—H9A	108.7
C7—N1—C8	118.1 (3)	C8—C9—H9B	108.7
C7—N1—Cu1	126.5 (2)	N2—C9—H9B	108.7
C8—N1—Cu1	115.4 (2)	H9A—C9—H9B	107.6
C10—N2—C12	119.0 (4)	C11—C10—N2	117.0 (5)
C10—N2—C9	107.4 (4)	C11—C10—H10A	108.0
C12—N2—C9	106.6 (3)	N2—C10—H10A	108.0
C10—N2—Cu1	110.7 (3)	C11—C10—H10B	108.0
C12—N2—Cu1	110.0 (2)	N2—C10—H10B	108.0
C9—N2—Cu1	101.6 (2)	H10A—C10—H10B	107.3
C14—N3—Cu1	159.8 (3)	C10—C11—H11A	109.5
O1—C1—C2	125.2 (3)	C10—C11—H11B	109.5
O1—C1—C6	119.2 (3)	H11A—C11—H11B	109.5
C2—C1—C6	115.6 (3)	C10—C11—H11C	109.5
C3—C2—C1	120.5 (3)	H11A—C11—H11C	109.5
C3—C2—C7	117.2 (3)	H11B—C11—H11C	109.5
C1—C2—C7	122.3 (3)	N2—C12—C13	117.3 (3)
C4—C3—C2	120.4 (3)	N2—C12—H12A	108.0
C4—C3—H3	119.8	C13—C12—H12A	108.0
C2—C3—H3	119.8	N2—C12—H12B	108.0
C3—C4—C5	121.0 (3)	C13—C12—H12B	108.0
C3—C4—Cl2	120.3 (3)	H12A—C12—H12B	107.2
C5—C4—Cl2	118.7 (3)	C12—C13—H13A	109.5
C6—C5—C4	119.0 (3)	C12—C13—H13B	109.5
C6—C5—H5	120.5	H13A—C13—H13B	109.5
C4—C5—H5	120.5	C12—C13—H13C	109.5
C5—C6—C1	123.4 (3)	H13A—C13—H13C	109.5
C5—C6—Cl1	119.2 (3)	H13B—C13—H13C	109.5
C1—C6—Cl1	117.4 (3)	N3—C14—S1	177.6 (3)
N1—C7—C2	125.3 (3)		

Symmetry codes: (i) −*x*, −*y*, −*z*.

Fig. 1


Cl2
C4
C5
C6
Cl1
C3
C1
C2
O1
C7
N1
Cu1
C8
C9
N2
C12
C10
C13
C11
N3
C14
S1