Crystal data

$[Cu(NCS)(C_{14}H_{32}N_4)]ClO_4$
$M_r = 477.50$
Monoclinic
$P2_1/c$
$a = 8.9245 (10)$ Å
$b = 14.9611 (10)$ Å
$c = 15.7331 (13)$ Å
$\beta = 96.184 (10)°$
$V = 2088.5 (3)$ Å^3
$Z = 4$
$D_x = 1.519$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 22 reflections
$\theta = 5.45–15.74°$
$\mu = 1.30$ mm^{-1}
$T = 293$ K
Cubic
0.25 × 0.13 × 0.12 mm
Light blue

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (North *et al.*, 1968)
$T_{min} = 0.703$, $T_{max} = 0.867$
3926 measured reflections
3675 independent reflections

2514 reflections with $I > 1.5\sigma(I)$
$R_{int} = 0.019$
$\theta_{max} = 24.93°$
$h = -10 \rightarrow 10$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 18$
3 standard reflections
frequency: 60 min
intensity decay: 1.0%

Refinement

Refinement on F
$R = 0.041$
$wR = 0.052$
$S = 1.13$
2513 reflections
245 parameters
H atoms not refined
$w = 1/[\sigma^2(F) + 0.001F^2]$
$(\Delta/\sigma)_{max} = 0.002$

$\Delta\rho_{max} = 0.39$ e Å^{-3}
$\Delta\rho_{min} = -0.28$ e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: $8 (2) \times 10^3$
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Cu—N1	2.080 (4)	Cu—N4	2.097 (4)
Cu—N2	2.094 (4)	Cu—N5	2.116 (4)
Cu—N3	2.143 (4)	S—C15	1.628 (5)
N1—Cu—N2	85.45 (15)	Cu—N1—C3	106.4 (3)
N1—Cu—N3	145.46 (15)	Cu—N2—C4	104.4 (3)
N1—Cu—N4	92.40 (15)	Cu—N2—C5	115.3 (3)
N1—Cu—N5	114.57 (16)	Cu—N2—C6	112.1 (3)
N2—Cu—N3	92.80 (16)	Cu—N3—C8	112.1 (3)
N2—Cu—N4	174.37 (15)	Cu—N3—C9	114.7 (3)
N2—Cu—N5	93.53 (15)	Cu—N3—C10	103.8 (3)
N3—Cu—N4	86.02 (15)	Cu—N4—C11	103.0 (3)
N3—Cu—N5	99.98 (16)	Cu—N4—C12	115.7 (3)
N4—Cu—N5	92.09 (16)	Cu—N4—C13	112.8 (3)
Cu—N1—C1	114.0 (3)	Cu—N5—C15	144.6 (4)
Cu—N1—C2	111.2 (3)	S—C15—N5	178.9 (4)

Data reduction: *NRCVAX DATRD2* (Gabe *et al.*, 1989). Program(s) used to solve structure: *NRCVAX SOLVER*. Program(s) used to refine structure: *NRCVAX LSTSQ*. Molecular graphics: *ORTEP*II (Johnson, 1976) in *NRCVAX*. Software used to prepare material for publication: *NRCVAX TABLES*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: OA1047). Services for accessing these data are described at the back of the journal.

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Bis[*N*-(2-bromo-4-methylphenyl)-naphthaldiminato]copper(II)

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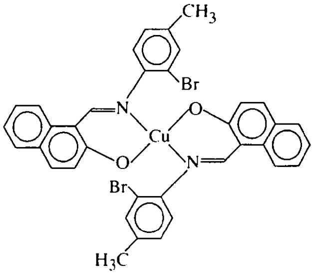
Abstract

The title compound, bis[1-(2-bromo-4-methylphenyliminomethyl)-2-naphtholato-*N*,*O*]copper(II), $[Cu(C_{18}H_{13}BrNO)_2]$, has crystallographic inversion symmetry. The Cu^{II} ion shows a pseudo-square-planar coordination. The Cu—N and Cu—O distances are 1.990 (4) and 1.880 (4) Å, respectively. The Cu^{II} ion is in an approximate octahedral environment if bromine is included in

the description, with a non-bonded Cu···Br distance of 3.296 (1) Å.

Comment

Because of their preparative accessibility and structural variability, a great number of Schiff base complexes of copper have been the subject of extensive studies (Garnovskii *et al.*, 1993). Despite this, there are few reported X-ray crystal structures of naphthalenic Schiff base–copper compounds (Acevedo-Arauz *et al.*, 1992). We report here the results of the reaction of copper(II) with the ligand *N*-(2-bromo-4-methylphenyl)naphthaldimine, which produced the title compound, (I).



(I)

The title molecule (Fig. 1) has crystallographic inversion symmetry. The coordination of the Cu^{II} ion is necessarily planar by symmetry. A strictly planar or slightly distorted *trans* configuration is characteristic for transition metal complexes of Cu^{II} with a CuN_2O_2 coordination sphere (Garnovskii *et al.*, 1993). In the title compound, the Cu^{II} ion is bonded to the O and N donor atoms of the two ligand molecules in a *trans* arrangement. The Cu—N and Cu—O distances are 1.990 (4) and 1.880 (4) Å, respectively. The O—Cu—N angle is 90.2 (2)°. The Cu—N distances range from 1.97 to 2.01 Å, while the Cu—O distances have values between 1.87 and 1.92 Å in (1,2-naphthaldiminato)copper complexes (Clark *et al.*, 1975, 1977; Martin & Waters, 1973; Shnulin *et al.*, 1978; Acevedo-Arauz *et al.*, 1992). The Cu—N and Cu—O values in (I) agree with those in other naphthalenic complexes. The Cu^{II} ion is in a pseudo-octahedral environment if bromine is included in the description. The non-bonded Cu···Br distance is 3.296 (1) Å. The title molecule is not planar; the Cu^{II} ion lies 0.238 (2) Å below the plane formed by the Cu1, O1, C1, C10, C11 and N1 atoms. This plane is inclined at an angle of 18.6 (1)° with respect to the coordination plane. The two Schiff base moieties, *A* (O1, C1–C11) and *B* (Br, N1, O2, C12–C18), are inclined at an angle of 43.0 (1)° with respect to one another, which is mainly the twist about N1—C12 [54.3 (7)°]. The N atom shows sp^2 hybridization, with a bond-angle sum of 359.5 (4)°.

Discrete monomeric molecules are held together in the crystal by van der Waals interactions. The minimum distances between two non-H atoms in neigh-

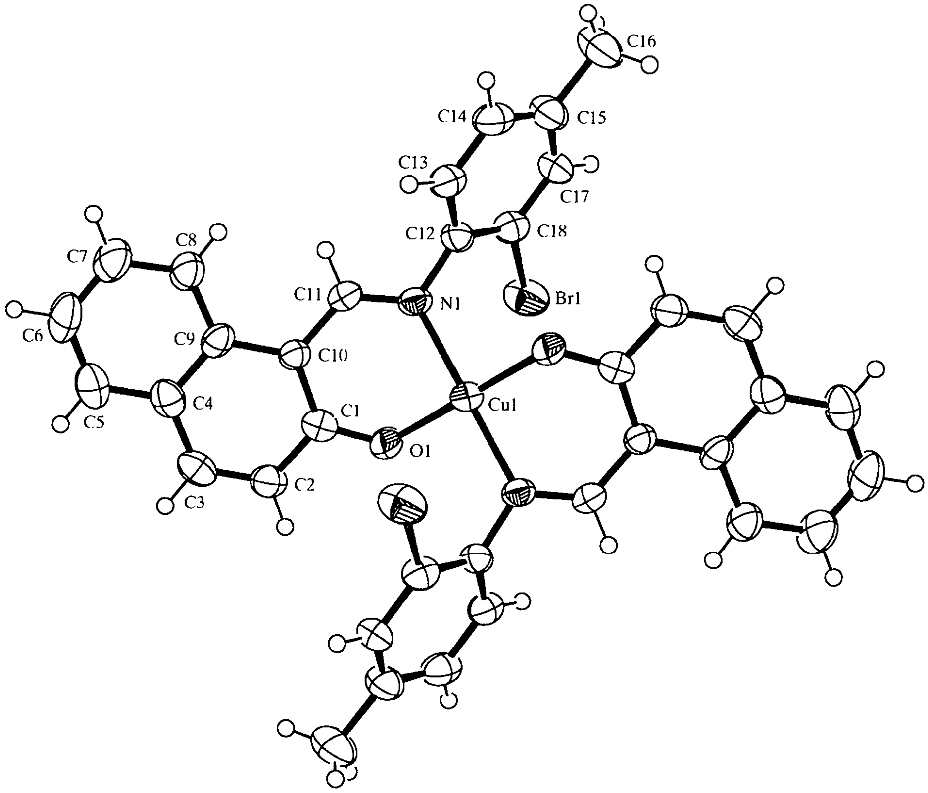


Fig. 1. The molecular structure of (I) showing 50% probability displacement ellipsoids (*ORTEP*II; Johnson, 1976).

bouring molecules are C8···O1, C8···C13, C9···C13 and O1···C7 with the values of 3.400 (7), 3.466 (9), 3.483 (9) and 3.518 (8) Å, respectively.

Experimental

For the preparation of (I), solutions of 2-hydroxynaphthalene-1-carbaldehyde (0.50 mmol) and 2-bromo-4-methylaniline (0.50 mmol) in 75 ml acetonitrile, and $[Cu(O_2CCH_3)_2].4H_2O$ (0.25 mmol) in 40 ml methanol were heated to boiling, mixed and refluxed for 3 d. Crystals of the product were obtained on cooling.

Crystal data

$[Cu(C_{18}H_{13}BrNO)_2]$
$M_r = 741.95$
Monoclinic
$P2_1/c$
$a = 9.255\,(1)$ Å
$b = 12.524\,(2)$ Å
$c = 13.538\,(2)$ Å
$\beta = 104.32\,(1)^\circ$
$V = 1520.4\,(4)$ Å^3
$Z = 2$
$D_x = 1.621$ Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
$\lambda = 0.71069$ Å
Cell parameters from 20 reflections
$\theta = 4.12–14.43^\circ$
$\mu = 3.383$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.28 × 0.18 × 0.10 mm
Purple–red

Data collection

Enraf–Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: ψ scan (*MolEN*; Fair, 1990)
$T_{\min} = 0.496$, $T_{\max} = 0.713$
2762 measured reflections
2760 independent reflections
1866 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.018$
$\theta_{\max} = 26.14^\circ$
$h = 0 \rightarrow 11$
$k = 0 \rightarrow 15$
$l = -16 \rightarrow 16$
3 standard reflections frequency: 120 min intensity decay: 3.2%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.056$
$wR(F^2) = 0.097$
$S = 1.208$
2760 reflections
196 parameters
H-atom parameters were constrained to parent sites
$w = 1/[\sigma^2(F_o^2) + 3.7194P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.491$ e Å^{-3}
$\Delta\rho_{\min} = -0.326$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

Cu1—O1$^{\rm i}$	1.880 (4)	N1—C11	1.295 (6)
Cu1—N1$^{\rm i}$	1.990 (4)	N1—C12	1.434 (6)
Br1—C18	1.900 (6)	O1—C1	1.293 (6)
O1—Cu1—N1$^{\rm i}$	90.2 (2)	O1—C1—C2	116.0 (5)
C11—N1—C12	118.2 (4)	N1—C11—C10	126.2 (5)
C11—N1—Cu1	122.3 (4)	C13—C12—N1	122.1 (5)
C12—N1—Cu1	119.0 (3)	C18—C12—N1	120.9 (5)
C1—O1—Cu1	124.8 (3)	C17—C18—Br1	118.9 (5)
O1—C1—C10	124.8 (5)	C12—C18—Br1	119.3 (4)

Symmetry code: (i) $-x, -y, -z$.

The title structure was solved by direct phase determination. The parameters of the complete structure could be refined by full-matrix anisotropic least squares. All phenyl rings were refined without constraints. Distance and angle values in the rings show no significant differences from those of an ideal benzene ring. All H-atom positions were calculated using a riding model with fixed $U_{\rm iso}$ values in all refinements.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994). Cell refinement: *MolEN* (Fair, 1990). Data reduction: *MolEN*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *ORTEP*II (Johnson, 1976). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: AB1532). Services for accessing these data are described at the back of the journal.

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