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Bis(5-bromo-1*H*-indole-3-carbaldehyde 2-nitrobenzoylhydrazonato-κ^2N,O)bis(*N*,*N*-dimethylformamide-κO)copper(II)

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The Cu atom in the title compound, $[Cu(C_{16}H_{10}BrN_4O_3)_2(C_3H_7NO)_2]$, lies on a special position of $\overline{1}$ site symmetry in a grossly elongated CuN_2O_4 octahedral geometry [$Cu\cdots O_{DMF}$ = 3.014 (4) Å]. The Cu atom is also *N*,*O*-chelated by the hydrazonate ligand. An $N-H\cdots O$ hydrogen bond helps to consolidate the crystal packing.

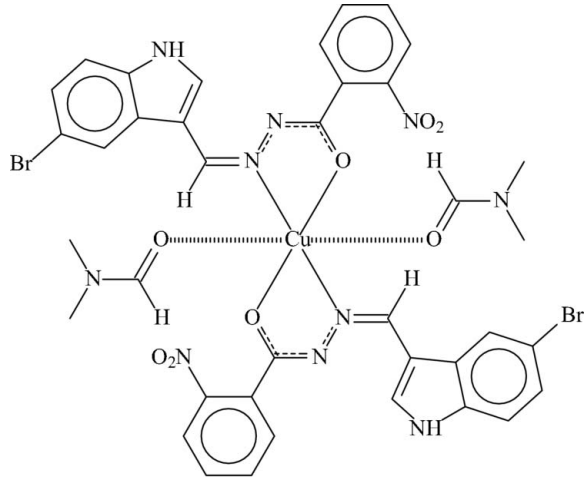
Key indicators

Single-crystal X-ray study
T = 295 K
Mean σ(C–C) = 0.006 Å
R factor = 0.055
wR factor = 0.180
Data-to-parameter ratio = 13.9

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

Comment

A previous report (Ali *et al.*, 2005*a*) described the crystal structure of the Schiff base that is synthesized by condensing 5-bromoindole-3-carbaldehyde with 2-nitrobenzoylhydrazone. In the title compound, (I) (Fig. 1), two of these deprotonated Schiff bases chelate to copper (site symmetry $\overline{1}$) in a square-planar geometry (Table 1). However, the O atom of the DMF molecule lies at a distance of 3.014 (4) Å from the Cu atom; thus, the copper coordination geometry can also be regarded as grossly distorted octahedral. The molecules of (I) are linked by an $N-H\cdots O_{DMF}$ hydrogen bond (Table 2) into a chain.



(I)

The nickel derivative of 5-bromoindole-3-carbaldehyde benzoylhydrazone was recrystallized from pyridine to afford the bis-pyridine adduct; the geometry of the metal atom coordination is an essentially regular octahedron, and the pyridine N atoms are *cis* to each other (Ali *et al.*, 2005*b*).

Experimental

5-Bromoindole-3-carboxaldehyde was condensed with 2-nitrobenzoylhydrazide to form the Schiff base 5-bromo-1*H*-indole-3-carbaldehyde 2-nitrobenzoylhydrazone (Ali *et al.*, 2005*b*). This

reactant (0.40 g, 1.0 mmol) and copper(II) acetate dihydrate (0.11 g, 0.5 mmol) were heated in ethanol (50 ml) for several hours. The solid that separated from solution was purified by recrystallization from DMF. Green crystals of (I) were isolated after two weeks.

Crystal data

$[Cu(C_{16}H_{10}BrN_4O_3)_2(C_3H_7NO)_2]$
M_r = 982.11
Triclinic, $P\overline{1}$
a = 8.643 (5) Å
b = 9.102 (5) Å
c = 14.302 (9) Å
α = 99.84 (2)°
β = 99.27 (1)°
γ = 106.43 (1)°
V = 1037 (1) Å^3
Z = 1
D_x = 1.573 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 3209 reflections
θ = 2.4–28.0°
μ = 2.52 mm^{-1}
T = 295 (2) K
Block, green
0.62 × 0.44 × 0.15 mm

Data collection

Rigaku Mercury CCD diffractometer
ω scans
Absorption correction: multi-scan (*CRYSTALCLEAR*; Rigaku/MSC, 2005)
T_{min} = 0.114, T_{max} = 0.686
17658 measured reflections
3797 independent reflections
2915 reflections with $I > 2\sigma(I)$
R_{int} = 0.050
θ_{max} = 25.5°
$h = -10 \rightarrow 10$
$k = -10 \rightarrow 11$
$l = -17 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.055
$wR(F^2)$ = 0.180
S = 1.10
3797 reflections
274 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.1064P)^2 + 0.1574P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.78 e Å^{-3}
$\Delta\rho_{min}$ = −0.64 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.930 (3)	Cu1—N3	1.937 (3)
Cu1—O4	3.014 (4)		
O1—Cu1—O4	85.0 (1)	N3—Cu1—O4	88.4 (1)
O1—Cu1—N3	81.8 (1)	N3^i—Cu1—O4	91.7 (1)
O1—Cu1—N3^i	98.2 (1)		

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

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N4—H4n···O4^{ii}	0.85 (1)	1.95 (2)	2.777 (4)	163 (5)

Symmetry code: (ii) $x, y+1, z$.

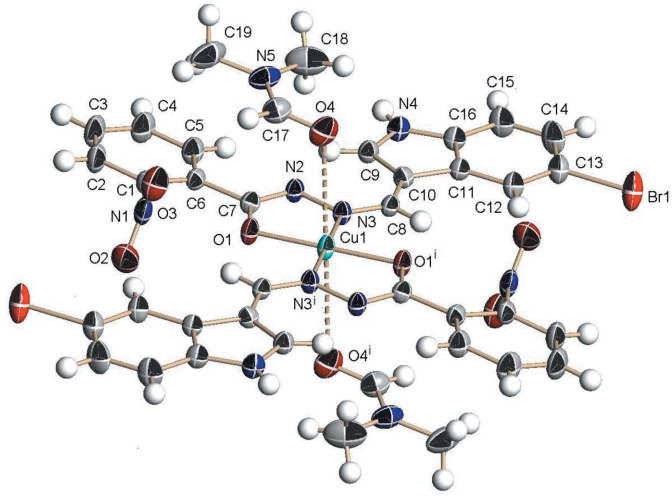


Figure 1
View of (I), showing 30% displacement ellipsoids (arbitrary spheres for the H atoms). The long Cu···O_{DMF} interaction is indicated by a dashed line [symmetry code: (i) $1-x, 1-y, 1-x$].

The carbon-bound H atoms were positioned geometrically (C—H = 0.93 or 0.96 Å) and refined as riding, with U_{iso}(H) = 1.2U_{eq}(C) or 1.5U_{eq}(methyl). The methyl groups were rotated to fit the electron density. The nitrogen-bound H atom was located in a difference Fourier map and refined with a distance restraint of N—H = 0.85 (1) Å; the U_{iso} value was freely refined.

Data collection: *CRYSTALCLEAR* (Rigaku/MSC, 2005); cell refinement: *CRYSTALCLEAR*; data reduction: *CRYSTALCLEAR*; program(s) used to solve structure: *CrystalStructure* (Rigaku/MSC, 2005); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97*.

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References

Ali, H. M., Abdul Halim, S. N. & Ng, S. W. (2005*a*). *Acta Cryst.* E**61**, o2308–o2309.
Ali, H. M., Abdul Halim, S. N. & Ng, S. W. (2005*b*). *Acta Cryst.* E**61**, m1682–m1683.
Johnson, C. K. (1976). *ORTEPII*. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Rigaku/MSC (2005). *CRYSTALCLEAR* and *CrystalStructure*. Rigaku/MSC Inc., 9009 New Trails Drive, The Woodlands, TX77381-5209, USA.
Sheldrick, G. M. (1997). *SHELXL97*. University of Göttingen, Germany.