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Bis{benzyl *N*′-[(1*H*-indol-3-yl)methylene]dithiocarbazato-κ^2N',S}copper(II) *N*,*N*-dimethylformamide disolvate

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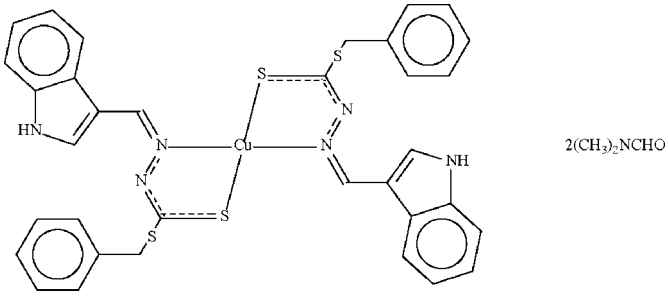
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(C–C) = 0.003 Å; R factor = 0.031; wR factor = 0.081; data-to-parameter ratio = 17.9.

In the structure of $[Cu(C_{17}H_{14}N_3S_2)_2]\cdot 2C_3H_7NO$, the Cu atom (site symmetry $\overline{1}$) is *N*,*S*-chelated by the two deprotonated Schiff-base anions that define a distorted square-planar geometry. An N—H···O hydrogen bond links the mononuclear complex to the DMF solvent molecules.

Related literature

For the Schiff base ligand, see: Khaledi *et al.* (2008*b*). For the isostructural nickel analog, see: Khaledi *et al.* (2008*a*).



Experimental

Crystal data

$[Cu(C_{17}H_{14}N_3S_2)_2]\cdot 2C_3H_7NO$
M_r = 858.60
Monoclinic, $P2_1/c$
a = 10.4461 (2) Å
b = 20.0882 (3) Å
c = 10.8333 (2) Å
β = 118.366 (1)°
V = 2000.34 (6) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 0.80 mm^{-1}
T = 100 (2) K
0.25 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.825, T_{max} = 0.961
18600 measured reflections
4594 independent reflections
3837 reflections with $I > 2\sigma(I)$
R_{int} = 0.035

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.04
4594 reflections
256 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N3—H3N···O1	0.88 (2)	1.87 (2)	2.742 (2)	175 (2)

Data collection: *APEX2* (Bruker, 2007); cell refinement: *SAINT* (Bruker, 2007); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2009).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: TK2349).

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supporting information

Acta Cryst. (2009). E**65**, m139 [doi:10.1107/S1600536808043808]

Bis{benzyl *N*′-[(1*H*-indol-3-yl)methylene]dithiocarbazato-κ^2N',S}copper(II) *N*,*N*-dimethylformamide disolvate

Hamid Khaledi, Hapipah Mohd Ali and Seik Weng Ng

S1. Experimental

Benzyl (1*H*-indol-2-ylmethylene)hydrazinecarbodithioate (Khaledi *et al.*, 2008*b*) (1 mmol, 0.33 g) was dissolved in ethanol (30 ml). To the clear solution was added an ethanol solution (10 ml) containing 1 mmol (0.09 g) of copper chloride dihydrate. The mixture was heated for an hour. The product that separated was recrystallized from DMF.

S2. Refinement

The C-bound H atoms were placed at calculated positions (C–H 0.95–0.99 Å) and were treated as riding on their parent C atoms, with *U*(H) set to 1.2–1.5 times $U_{eq}(C)$. The amino H-atom was located in a difference Fourier map, and was refined with a distance restraint of N–H 0.88±0.01 Å; its temperature factor was freely refined.

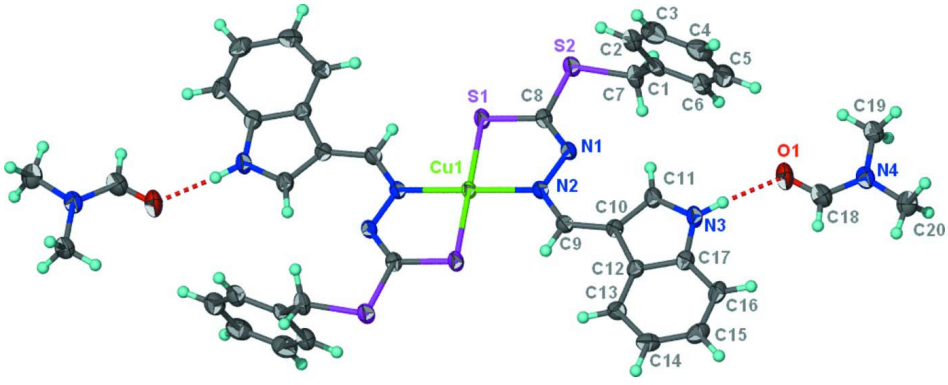

Figure 1

Thermal ellipsoid plot (Barbour, 2001) of $Cu(C_{17}H_{14}N_3)\cdot 2DMF$ at the 70% probability level. Hydrogen atoms are drawn as spheres of arbitrary radius.

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c = 10.8333 (2) Å
β = 118.366 (1)°
V = 2000.34 (6) Å^3
Z = 2
$F(000)$ = 894
D_x = 1.425 Mg m^{-3}
Mo $K\alpha$ radiation, λ = 0.71073 Å
μ = 0.80 mm^{-1}
T = 100 K
Irregular block, brown
0.25 × 0.15 × 0.05 mm

Data collection

Bruker SMART APEX diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
ω scans
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.825, T_{max} = 0.961
18600 measured reflections
4594 independent reflections
3837 reflections with $I > 2\sigma(I)$
R_{int} = 0.035
θ_{max} = 27.5°, θ_{min} = 2.0°
h = −13→13
k = −25→26
l = −14→14

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.081
S = 1.04
4594 reflections
256 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 atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0382P)^2 + 0.7321P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 0.36 e Å^{-3}
$\Delta\rho_{min}$ = −0.36 e Å^{-3}

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.5000	0.5000	0.5000	0.01678 (8)
O1	0.8350 (2)	0.56467 (8)	−0.10421 (17)	0.0526 (5)
S1	0.51561 (5)	0.61250 (2)	0.51610 (5)	0.02414 (11)
S2	0.61468 (5)	0.70543 (2)	0.37409 (5)	0.02345 (11)
N1	0.60380 (15)	0.57545 (7)	0.32793 (14)	0.0191 (3)
N2	0.56823 (15)	0.51211 (7)	0.35752 (15)	0.0193 (3)
N3	0.72111 (17)	0.49444 (7)	0.03797 (16)	0.0234 (3)
H3N	0.756 (3)	0.5192 (11)	−0.006 (2)	0.040 (6)*
N4	0.91692 (17)	0.57480 (8)	−0.26314 (16)	0.0267 (3)
C1	0.81749 (19)	0.68443 (8)	0.27140 (19)	0.0223 (4)
C2	0.9316 (2)	0.68264 (9)	0.4074 (2)	0.0282 (4)
H2	0.9149	0.6937	0.4838	0.034*
C3	1.0699 (2)	0.66472 (10)	0.4321 (2)	0.0338 (4)
H3	1.1469	0.6628	0.5258	0.041*
C4	1.0974 (2)	0.64964 (10)	0.3226 (2)	0.0340 (5)
H4	1.1926	0.6376	0.3406	0.041*
C5	0.9852 (2)	0.65223 (10)	0.1870 (2)	0.0315 (4)
H5	1.0030	0.6423	0.1108	0.038*
C6	0.8463 (2)	0.66931 (9)	0.1618 (2)	0.0261 (4)
H6	0.7695	0.6707	0.0680	0.031*
C7	0.6635 (2)	0.70369 (9)	0.23471 (19)	0.0245 (4)
H7A	0.5968	0.6724	0.1620	0.029*
H7B	0.6446	0.7485	0.1915	0.029*
C8	0.58034 (17)	0.62208 (8)	0.39595 (17)	0.0188 (3)

C9	0.58804 (18)	0.46384 (8)	0.28764 (18)	0.0204 (3)
H9	0.5641	0.4209	0.3071	0.024*
C10	0.63940 (18)	0.46470 (8)	0.18750 (17)	0.0201 (3)
C11	0.68033 (19)	0.51762 (9)	0.13067 (19)	0.0229 (4)
H11	0.6796	0.5632	0.1538	0.027*
C12	0.65845 (18)	0.40510 (8)	0.12243 (17)	0.0196 (3)
C13	0.63965 (19)	0.33709 (9)	0.13551 (18)	0.0234 (4)
H13	0.6062	0.3216	0.1980	0.028*
C14	0.6707 (2)	0.29284 (9)	0.0556 (2)	0.0276 (4)
H14	0.6595	0.2464	0.0645	0.033*
C15	0.7185 (2)	0.31520 (9)	−0.0383 (2)	0.0285 (4)
H15	0.7380	0.2837	−0.0926	0.034*
C16	0.73753 (19)	0.38186 (9)	−0.05314 (18)	0.0250 (4)
H16	0.7690	0.3972	−0.1173	0.030*
C17	0.70879 (18)	0.42596 (9)	0.02967 (18)	0.0211 (3)
C18	0.8710 (3)	0.54041 (11)	−0.1878 (2)	0.0395 (5)
H18	0.8652	0.4934	−0.1989	0.047*
C19	0.9269 (2)	0.64686 (9)	−0.2527 (2)	0.0294 (4)
H19A	0.9223	0.6613	−0.1685	0.044*
H19B	0.8460	0.6666	−0.3358	0.044*
H19C	1.0193	0.6613	−0.2467	0.044*
C20	0.9516 (2)	0.54244 (10)	−0.3636 (2)	0.0314 (4)
H20A	0.9441	0.4941	−0.3573	0.047*
H20B	1.0510	0.5541	−0.3427	0.047*
H20C	0.8832	0.5573	−0.4587	0.047*

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.01726 (15)	0.01781 (15)	0.01939 (15)	0.00000 (11)	0.01205 (12)	0.00001 (11)
O1	0.0872 (13)	0.0476 (10)	0.0468 (9)	−0.0192 (9)	0.0511 (10)	−0.0044 (7)
S1	0.0345 (3)	0.0189 (2)	0.0316 (2)	−0.00003 (17)	0.0260 (2)	−0.00067 (17)
S2	0.0308 (2)	0.0175 (2)	0.0305 (2)	0.00151 (17)	0.0214 (2)	0.00143 (17)
N1	0.0217 (7)	0.0178 (7)	0.0208 (7)	−0.0002 (5)	0.0126 (6)	0.0020 (6)
N2	0.0222 (7)	0.0179 (7)	0.0214 (7)	−0.0007 (5)	0.0134 (6)	0.0007 (5)
N3	0.0284 (8)	0.0236 (8)	0.0266 (8)	0.0016 (6)	0.0198 (7)	0.0027 (6)
N4	0.0282 (8)	0.0283 (8)	0.0251 (8)	−0.0006 (6)	0.0138 (7)	0.0045 (6)
C1	0.0268 (9)	0.0148 (8)	0.0302 (9)	−0.0002 (7)	0.0175 (8)	0.0050 (7)
C2	0.0309 (10)	0.0290 (10)	0.0281 (9)	−0.0024 (8)	0.0168 (8)	0.0022 (8)
C3	0.0247 (10)	0.0349 (11)	0.0346 (11)	−0.0042 (8)	0.0082 (9)	0.0018 (9)
C4	0.0256 (10)	0.0290 (10)	0.0506 (13)	−0.0022 (8)	0.0207 (10)	−0.0036 (9)
C5	0.0329 (11)	0.0303 (10)	0.0405 (11)	−0.0016 (8)	0.0249 (10)	−0.0031 (8)
C6	0.0285 (9)	0.0252 (9)	0.0290 (9)	−0.0006 (7)	0.0172 (8)	0.0027 (7)
C7	0.0291 (9)	0.0236 (9)	0.0274 (9)	0.0054 (7)	0.0187 (8)	0.0081 (7)
C8	0.0158 (8)	0.0215 (8)	0.0196 (8)	0.0016 (6)	0.0090 (7)	0.0025 (6)
C9	0.0223 (9)	0.0182 (8)	0.0240 (9)	−0.0011 (6)	0.0137 (7)	−0.0003 (7)
C10	0.0214 (8)	0.0197 (8)	0.0212 (8)	−0.0004 (6)	0.0118 (7)	−0.0010 (7)
C11	0.0262 (9)	0.0219 (8)	0.0272 (9)	0.0022 (7)	0.0182 (8)	0.0003 (7)

C12	0.0178 (8)	0.0226 (8)	0.0197 (8)	0.0000 (6)	0.0100 (7)	−0.0026 (7)
C13	0.0223 (9)	0.0239 (9)	0.0259 (9)	−0.0034 (7)	0.0129 (8)	−0.0027 (7)
C14	0.0238 (9)	0.0227 (9)	0.0355 (10)	−0.0022 (7)	0.0135 (8)	−0.0060 (8)
C15	0.0254 (9)	0.0303 (10)	0.0310 (10)	0.0022 (8)	0.0143 (8)	−0.0098 (8)
C16	0.0223 (9)	0.0340 (10)	0.0220 (9)	0.0023 (7)	0.0131 (8)	−0.0034 (7)
C17	0.0192 (8)	0.0237 (9)	0.0210 (8)	0.0025 (7)	0.0101 (7)	−0.0001 (7)
C18	0.0543 (14)	0.0337 (11)	0.0362 (11)	−0.0107 (10)	0.0262 (11)	0.0016 (9)
C19	0.0338 (11)	0.0283 (10)	0.0271 (10)	−0.0031 (8)	0.0154 (9)	0.0030 (8)
C20	0.0308 (10)	0.0354 (11)	0.0312 (10)	0.0037 (8)	0.0173 (9)	0.0028 (8)

Geometric parameters (Å, º)

Cu1—N2	1.9987 (14)	C5—H5	0.9500
Cu1—N2^{i}	1.9987 (14)	C6—H6	0.9500
Cu1—S1	2.2666 (4)	C7—H7A	0.9900
Cu1—S1^{i}	2.2666 (4)	C7—H7B	0.9900
O1—C18	1.234 (3)	C9—C10	1.421 (2)
S1—C8	1.7392 (17)	C9—H9	0.9500
S2—C8	1.7519 (17)	C10—C11	1.394 (2)
S2—C7	1.8092 (17)	C10—C12	1.450 (2)
N1—C8	1.285 (2)	C11—H11	0.9500
N1—N2	1.4048 (18)	C12—C17	1.401 (2)
N2—C9	1.306 (2)	C12—C13	1.397 (2)
N3—C11	1.347 (2)	C13—C14	1.382 (2)
N3—C17	1.381 (2)	C13—H13	0.9500
N3—H3N	0.88 (2)	C14—C15	1.403 (3)
N4—C18	1.321 (2)	C14—H14	0.9500
N4—C19	1.452 (2)	C15—C16	1.374 (3)
N4—C20	1.454 (2)	C15—H15	0.9500
C1—C6	1.390 (2)	C16—C17	1.391 (2)
C1—C2	1.387 (3)	C16—H16	0.9500
C1—C7	1.512 (2)	C18—H18	0.9500
C2—C3	1.386 (3)	C19—H19A	0.9800
C2—H2	0.9500	C19—H19B	0.9800
C3—C4	1.380 (3)	C19—H19C	0.9800
C3—H3	0.9500	C20—H20A	0.9800
C4—C5	1.378 (3)	C20—H20B	0.9800
C4—H4	0.9500	C20—H20C	0.9800
C5—C6	1.386 (3)		

N2—Cu1—N2^{i}	180.000 (1)	S1—C8—S2	112.60 (9)
N2—Cu1—S1	84.21 (4)	N2—C9—C10	130.93 (16)
N2^{i}—Cu1—S1	95.79 (4)	N2—C9—H9	114.5
N2—Cu1—S1^{i}	95.79 (4)	C10—C9—H9	114.5
N2^{i}—Cu1—S1^{i}	84.21 (4)	C11—C10—C9	130.77 (16)
S1—Cu1—S1^{i}	180.0	C11—C10—C12	105.86 (15)
C8—S1—Cu1	95.28 (6)	C9—C10—C12	123.36 (15)
C8—S2—C7	104.55 (8)	N3—C11—C10	109.74 (15)

C8—N1—N2	112.72 (13)	N3—C11—H11	125.1
C9—N2—N1	114.11 (14)	C10—C11—H11	125.1
C9—N2—Cu1	124.77 (12)	C17—C12—C13	118.93 (15)
N1—N2—Cu1	121.10 (10)	C17—C12—C10	106.66 (15)
C11—N3—C17	109.91 (15)	C13—C12—C10	134.40 (16)
C11—N3—H3N	124.5 (15)	C14—C13—C12	118.64 (16)
C17—N3—H3N	125.5 (15)	C14—C13—H13	120.7
C18—N4—C19	120.47 (17)	C12—C13—H13	120.7
C18—N4—C20	121.44 (17)	C13—C14—C15	121.20 (17)
C19—N4—C20	118.02 (15)	C13—C14—H14	119.4
C6—C1—C2	118.43 (17)	C15—C14—H14	119.4
C6—C1—C7	117.81 (16)	C16—C15—C14	121.23 (17)
C2—C1—C7	123.75 (16)	C16—C15—H15	119.4
C3—C2—C1	120.16 (18)	C14—C15—H15	119.4
C3—C2—H2	119.9	C15—C16—C17	117.19 (17)
C1—C2—H2	119.9	C15—C16—H16	121.4
C4—C3—C2	120.95 (18)	C17—C16—H16	121.4
C4—C3—H3	119.5	N3—C17—C16	129.38 (16)
C2—C3—H3	119.5	N3—C17—C12	107.84 (14)
C3—C4—C5	119.34 (18)	C16—C17—C12	122.79 (16)
C3—C4—H4	120.3	O1—C18—N4	125.0 (2)
C5—C4—H4	120.3	O1—C18—H18	117.5
C4—C5—C6	119.94 (18)	N4—C18—H18	117.5
C4—C5—H5	120.0	N4—C19—H19A	109.5
C6—C5—H5	120.0	N4—C19—H19B	109.5
C5—C6—C1	121.17 (18)	H19A—C19—H19B	109.5
C5—C6—H6	119.4	N4—C19—H19C	109.5
C1—C6—H6	119.4	H19A—C19—H19C	109.5
C1—C7—S2	118.16 (12)	H19B—C19—H19C	109.5
C1—C7—H7A	107.8	N4—C20—H20A	109.5
S2—C7—H7A	107.8	N4—C20—H20B	109.5
C1—C7—H7B	107.8	H20A—C20—H20B	109.5
S2—C7—H7B	107.8	N4—C20—H20C	109.5
H7A—C7—H7B	107.1	H20A—C20—H20C	109.5
N1—C8—S1	126.61 (13)	H20B—C20—H20C	109.5
N1—C8—S2	120.80 (13)		

N2—Cu1—S1—C8	−1.75 (7)	Cu1—N2—C9—C10	−177.95 (14)
N2^{i}—Cu1—S1—C8	178.25 (7)	N2—C9—C10—C11	−2.0 (3)
C8—N1—N2—C9	178.63 (15)	N2—C9—C10—C12	178.22 (17)
C8—N1—N2—Cu1	−3.25 (19)	C17—N3—C11—C10	0.1 (2)
S1—Cu1—N2—C9	−179.02 (14)	C9—C10—C11—N3	−179.57 (17)
S1^{i}—Cu1—N2—C9	0.98 (14)	C12—C10—C11—N3	0.2 (2)
S1—Cu1—N2—N1	3.07 (11)	C11—C10—C12—C17	−0.39 (19)
S1^{i}—Cu1—N2—N1	−176.93 (11)	C9—C10—C12—C17	179.40 (15)
C6—C1—C2—C3	−1.4 (3)	C11—C10—C12—C13	178.57 (19)
C7—C1—C2—C3	−179.70 (17)	C9—C10—C12—C13	−1.6 (3)
C1—C2—C3—C4	1.2 (3)	C17—C12—C13—C14	−0.4 (2)

C2—C3—C4—C5	−0.3 (3)	C10—C12—C13—C14	−179.23 (18)
C3—C4—C5—C6	−0.5 (3)	C12—C13—C14—C15	−0.8 (3)
C4—C5—C6—C1	0.4 (3)	C13—C14—C15—C16	0.7 (3)
C2—C1—C6—C5	0.6 (3)	C14—C15—C16—C17	0.6 (3)
C7—C1—C6—C5	179.01 (16)	C11—N3—C17—C16	179.22 (18)
C6—C1—C7—S2	166.51 (13)	C11—N3—C17—C12	−0.3 (2)
C2—C1—C7—S2	−15.1 (2)	C15—C16—C17—N3	178.73 (17)
C8—S2—C7—C1	−80.69 (15)	C15—C16—C17—C12	−1.8 (3)
N2—N1—C8—S1	1.3 (2)	C13—C12—C17—N3	−178.72 (15)
N2—N1—C8—S2	−179.21 (11)	C10—C12—C17—N3	0.43 (18)
Cu1—S1—C8—N1	0.81 (16)	C13—C12—C17—C16	1.7 (3)
Cu1—S1—C8—S2	−178.73 (8)	C10—C12—C17—C16	−179.15 (16)
C7—S2—C8—N1	5.62 (16)	C19—N4—C18—O1	−0.7 (3)
C7—S2—C8—S1	−174.81 (9)	C20—N4—C18—O1	−177.5 (2)
N1—N2—C9—C10	0.1 (3)		

Symmetry code: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N3—H3*N*···O1	0.88 (2)	1.87 (2)	2.742 (2)	175 (2)