Acta Crystallographica Section E
Structure Reports Online
ISSN 1600-5368

[2′-(5-Chloro-2-oxidobenzylidene)-benzenesulfonohydrazide-κ^2N,O]-[2′-(2-oxidobenzylidene)benzenesulfonohydrazide-κ^2N,O]copper(II)

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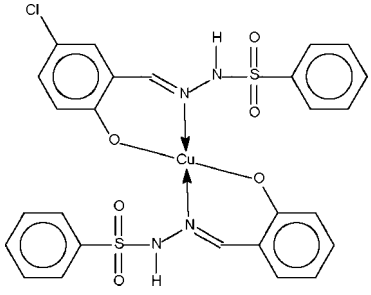
Received 2 November 2007; accepted 3 November 2007

Key indicators: single-crystal X-ray study; T = 106 K; mean σ(C–C) = 0.005 Å; disorder in main residue; R factor = 0.031; wR factor = 0.114; data-to-parameter ratio = 15.8.

The Cu^{II} atom (site symmetry $\bar{1}$) in the title compound, $[Cu(C_{13}H_{10}ClN_2O_3S)(C_{13}H_{11}N_2O_3S)]$, is N,O-chelated by the monoanionic ligands in a *trans*-CuN_2O_2 square-planar geometry. The 2′-(2-oxidobenzylidene)benzenesulfonohydrazide anion is disordered equallywith the chlorine-substituted 2′-(5-chloro-2-oxidobenzylidene)benzenesulfonohydrazide anion. An intermolecular N—H···O hydrogen bond helps to stabilize the crystal structure.

Related literature

For the structure of a related ligand, see: Ali *et al.* (2007).



Experimental

Crystal data

$[Cu(C_{13}H_{10}ClN_2O_3S)(C_{13}H_{11}N_2O_3S)]$
M_r = 648.61
Triclinic, $P\bar{1}$
a = 7.9801 (2) Å
b = 9.9993 (2) Å
c = 10.0823 (2) Å
α = 104.393 (1)°
β = 111.091 (1)°
γ = 104.635 (1)°
V = 672.96 (3) Å^3
Z = 1
Mo $K\alpha$ radiation
μ = 1.12 mm^{-1}
T = 106 (2) K
0.55 × 0.40 × 0.21 mm

Data collection

Bruker APEX-II diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.621, T_{max} = 0.800
6161 measured reflections
3026 independent reflections
2769 reflections with $I > 2\sigma(I)$
R_{int} = 0.016

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.031
$wR(F^2)$ = 0.114
S = 1.20
3026 reflections
191 parameters
1 restraint
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max}$ = 0.47 e Å^{-3}
$\Delta\rho_{min}$ = −0.37 e Å^{-3}

Table 1
Selected bond lengths (Å).

Cu1—O1	1.9062 (18)	Cu1—N1	1.9532 (17)

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2N···O1^{i}	0.87 (3)	2.13 (3)	2.723 (2)	125 (3)

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

Data collection: *APEX2* (Bruker, 2005); cell refinement: *SAINT* (Bruker, 2005); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *X-SEED* (Barbour, 2001); software used to prepare material for publication: *publCIF* (Westrip, 2007).

The authors thank the University of Canterbury, New Zealand, for the diffraction measurements, and the Science Fund (12–02-03–2031) and the Fundamental Research Grant Scheme (FP064/2006 A) for supporting this study.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB2629).

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supplementary materials

Acta Cryst. (2007). E**63**, m2937 [**doi:10.1107/S1600536807055663**]

[2'-(5-Chloro-2-oxidobenzylidene)benzenesulfonohydrazide-κ^2N,O][2'-(2-oxidobenzylidene)benzenesulfonohydrazide-κ^2N,O]copper(II)

H. M. Ali, J. Yusnita, M. R. Rizal and S. W. Ng

Comment

The title compound is a mixed-ligand compound in which the $C_{13}H_{11}O_3N_2S$ anion is disordered with respect to a $C_{13}H_{10}O_3N_2ClS$ anion. Although the synthesis had used 4-chlorobenzaldehyde as a starting material to prepare the Schiff base, this reagent is probably contaminated with an unknown quantity of benzaldehyde itself.

Experimental

Benzene sulfonylhydrazide (0.5 g, 0.3 mmol) and 4-chlorobenzaldehyde (0.5 g, 0.3 mmol) were dissolved in ethanol (50 ml). The reactants were heated under reflux for 1 h. The solvent was removed to give the Schiff base, which was purified by recrystallization from ethyl acetate. The organic compound (0.6 g, 2 mmol) dissolved in basified ethanol (50 ml) was heated with copper acetate (0.2 g m, 1 mmol) for 5 h. The solvent was removed and the product recrystallized from DMSO to yield golden blocks of (I).

Refinement

The refinement initally assumed full occupancy for the chlorine atom but the refinement gave a deep hole in its vicinity. The occupany was allowed to refine; as this refined to nearly 1/2, the occupancy was then set as half. The half-occupancy chlorine atom implies that (I) is a mixed ligand compound having a chlorine substitutent in 50% of the anions but none in the other 50%.

The carbon-bound H atoms were placed at calculated positions (C–H 0.95 Å), and were included in the refinement in the riding model approximation with U(H) set to 1.2U_{eq}(C). The amino hydrogen atom was located in a difference Fouier map, and was refined with a distance restraint of N–H 0.88±0.01 Å.

Figures

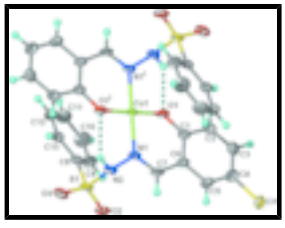


Fig. 1. **Figure 1**. View of the molecular structure of (I). Displacement ellipsoids are drawn at the 50% probability level, and H atoms are shown as spheres of arbitrary radii. The chlorine atom is statistically disordered with respect to a hydrogen atom and only one possible arrangement of these atoms is shown. The dashed lines denote the intramolecular hydrogen bond. Symmetry code (i) 1 – x, 1 – y, 1 – z.

[2'-(5-Chloro-2-oxidobenzylidene)benzenesulfonohydrazide- κ^2N,O][2'-(2-oxidobenzylidene)benzenesulfonohydrazide- κ^2N,O)]copper(II)

Crystal data

$[Cu(C_{13}H_{10}ClN_2O_3S)(C_{13}H_{11}N_2O_3S)]$	$Z = 1$
$M_r = 648.61$	$F_{000} = 331$
Triclinic, $P\overline{1}$	$D_x = 1.600$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 7.9801$ (2) Å	Cell parameters from 4328 reflections
$b = 9.9993$ (2) Å	$\theta = 2.3$–31.7º
$c = 10.0823$ (2) Å	$\mu = 1.12$ mm^{-1}
$\alpha = 104.393$ (1)º	$T = 106$ (2) K
$\beta = 111.091$ (1)º	Irregular block, gold
$\gamma = 104.635$ (1)º	0.55 × 0.40 × 0.21 mm
$V = 672.96$ (3) Å^3	

Data collection

Bruker APEX-II diffractometer	3026 independent reflections
Radiation source: medium-focus sealed tube	2769 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.016$
$T = 106$(2) K	$\theta_{max} = 27.5$º
φ and ω scans	$\theta_{min} = 2.3$º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -10 \rightarrow 10$
$T_{min} = 0.621$, $T_{max} = 0.800$	$k = -12 \rightarrow 12$
6161 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.031$	H atoms treated by a mixture of independent and constrained refinement
$wR(F^2) = 0.114$	$w = 1/[\sigma^2(F_o^2) + (0.0623P)^2 + 0.3245P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.20$	$(\Delta/\sigma)_{max} = 0.001$
3026 reflections	$\Delta\rho_{max} = 0.47$ e Å^{-3}
191 parameters	$\Delta\rho_{min} = -0.37$ e Å^{-3}
1 restraint	Extinction correction: none
Primary atom site location: structure-invariant direct methods	

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

	x	*y*	*z*	U_{iso}*/U_{eq}	Occ. (<1)
Cu1	0.5000	0.5000	0.5000	0.02500 (14)	
Cl1	0.1626 (2)	−0.10643 (15)	−0.20334 (14)	0.0409 (3)	0.50
S1	0.45362 (9)	0.19783 (6)	0.68120 (8)	0.03327 (17)	
O1	0.2978 (2)	0.41982 (17)	0.2953 (2)	0.0307 (4)	
N1	0.5095 (3)	0.3035 (2)	0.4831 (2)	0.0280 (4)	
N2	0.6053 (3)	0.2800 (2)	0.6200 (3)	0.0313 (4)	
H2N	0.691 (4)	0.363 (2)	0.695 (3)	0.042 (8)*	
O2	0.3354 (3)	0.05096 (19)	0.5676 (3)	0.0431 (5)	
O3	0.5800 (3)	0.2193 (2)	0.8345 (3)	0.0469 (5)	
C1	0.2593 (3)	0.2975 (3)	0.1829 (3)	0.0316 (5)	
C2	0.1386 (4)	0.2744 (3)	0.0309 (3)	0.0391 (6)	
H2	0.0805	0.3440	0.0118	0.047*	
C3	0.1027 (5)	0.1518 (3)	−0.0915 (3)	0.0448 (7)	
H3	0.0224	0.1390	−0.1934	0.054*	
C4	0.1844 (5)	0.0473 (3)	−0.0649 (4)	0.0478 (7)	
H4	0.1626	−0.0349	−0.1493	0.057*	0.50
C5	0.2947 (4)	0.0620 (3)	0.0803 (4)	0.0422 (6)	
H5	0.3449	−0.0122	0.0967	0.051*	
C6	0.3359 (4)	0.1868 (2)	0.2076 (3)	0.0325 (5)	
C7	0.4457 (3)	0.1906 (2)	0.3564 (3)	0.0319 (5)	
H7	0.4740	0.1051	0.3635	0.038*	
C8	0.3039 (3)	0.2999 (3)	0.6841 (3)	0.0313 (5)	
C9	0.1162 (4)	0.2416 (3)	0.5700 (4)	0.0416 (6)	
H9	0.0649	0.1455	0.4920	0.050*	
C10	0.0038 (4)	0.3275 (4)	0.5720 (4)	0.0526 (8)	
H10	−0.1263	0.2896	0.4945	0.063*	
C11	0.0797 (4)	0.4675 (4)	0.6855 (4)	0.0486 (7)	
H11	0.0015	0.5252	0.6848	0.058*	
C12	0.2683 (4)	0.5243 (3)	0.8000 (4)	0.0412 (6)	
H12	0.3194	0.6202	0.8781	0.049*	
C13	0.3824 (4)	0.4401 (3)	0.7998 (3)	0.0338 (5)	
H13	0.5123	0.4777	0.8777	0.041*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0256 (2)	0.01650 (19)	0.0427 (3)	0.01142 (15)	0.02107 (18)	0.01444 (16)
Cl1	0.0469 (7)	0.0369 (6)	0.0316 (6)	0.0084 (5)	0.0199 (5)	0.0059 (5)
S1	0.0386 (3)	0.0245 (3)	0.0532 (4)	0.0176 (2)	0.0279 (3)	0.0238 (3)
O1	0.0314 (8)	0.0208 (7)	0.0440 (10)	0.0122 (6)	0.0192 (7)	0.0128 (7)
N1	0.0286 (9)	0.0212 (9)	0.0464 (11)	0.0137 (7)	0.0237 (9)	0.0174 (8)
N2	0.0302 (10)	0.0247 (9)	0.0500 (13)	0.0148 (8)	0.0229 (10)	0.0193 (9)
O2	0.0498 (11)	0.0220 (8)	0.0711 (13)	0.0155 (8)	0.0365 (10)	0.0228 (9)
O3	0.0577 (12)	0.0452 (11)	0.0599 (13)	0.0313 (10)	0.0304 (11)	0.0362 (10)

C1	0.0313 (11)	0.0220 (10)	0.0476 (14)	0.0073 (9)	0.0247 (11)	0.0146 (10)
C2	0.0406 (13)	0.0291 (12)	0.0486 (15)	0.0070 (10)	0.0236 (12)	0.0172 (11)
C3	0.0509 (16)	0.0360 (14)	0.0426 (15)	0.0014 (12)	0.0267 (13)	0.0138 (12)
C4	0.0613 (18)	0.0286 (12)	0.0585 (18)	0.0068 (12)	0.0434 (16)	0.0101 (12)
C5	0.0540 (16)	0.0243 (11)	0.0586 (18)	0.0116 (11)	0.0406 (15)	0.0122 (12)
C6	0.0341 (12)	0.0198 (10)	0.0507 (15)	0.0083 (9)	0.0282 (11)	0.0126 (10)
C7	0.0330 (12)	0.0196 (10)	0.0563 (15)	0.0135 (9)	0.0300 (12)	0.0165 (10)
C8	0.0338 (12)	0.0271 (11)	0.0507 (14)	0.0154 (9)	0.0297 (11)	0.0225 (11)
C9	0.0356 (13)	0.0345 (13)	0.0595 (17)	0.0111 (11)	0.0297 (13)	0.0152 (12)
C10	0.0296 (13)	0.0598 (19)	0.074 (2)	0.0210 (13)	0.0277 (14)	0.0238 (17)
C11	0.0444 (15)	0.0522 (17)	0.077 (2)	0.0316 (14)	0.0429 (16)	0.0316 (16)
C12	0.0517 (16)	0.0337 (13)	0.0593 (17)	0.0220 (12)	0.0412 (14)	0.0201 (12)
C13	0.0374 (12)	0.0312 (12)	0.0470 (14)	0.0154 (10)	0.0280 (11)	0.0207 (11)

Geometric parameters (Å, °)

Cu1—O1^{i}	1.9062 (18)	C3—H3	0.9500
Cu1—O1	1.9062 (18)	C4—C5	1.356 (5)
Cu1—N1^{i}	1.9532 (17)	C4—H4	0.9500
Cu1—N1	1.9532 (17)	C5—C6	1.416 (4)
Cl1—C4	1.721 (3)	C5—H5	0.9500
S1—O2	1.430 (2)	C6—C7	1.424 (4)
S1—O3	1.434 (2)	C7—H7	0.9500
S1—N2	1.676 (2)	C8—C9	1.375 (4)
S1—C8	1.760 (2)	C8—C13	1.392 (4)
O1—C1	1.321 (3)	C9—C10	1.392 (4)
N1—C7	1.300 (3)	C9—H9	0.9500
N1—N2	1.427 (3)	C10—C11	1.383 (5)
N2—H2N	0.87 (3)	C10—H10	0.9500
C1—C2	1.406 (4)	C11—C12	1.383 (4)
C1—C6	1.425 (3)	C11—H11	0.9500
C2—C3	1.387 (4)	C12—C13	1.387 (3)
C2—H2	0.9500	C12—H12	0.9500
C3—C4	1.395 (5)	C13—H13	0.9500
O1^{i}—Cu1—O1	180.0	C3—C4—Cl1	126.0 (3)
O1^{i}—Cu1—N1^{i}	91.01 (8)	C5—C4—H4	119.7
O1—Cu1—N1^{i}	88.99 (8)	C3—C4—H4	119.7
O1^{i}—Cu1—N1	88.99 (8)	C4—C5—C6	120.7 (3)
O1—Cu1—N1	91.01 (8)	C4—C5—H5	119.6
N1^{i}—Cu1—N1	180.0	C6—C5—H5	119.6
O2—S1—O3	120.67 (12)	C5—C6—C7	117.2 (2)
O2—S1—N2	106.95 (12)	C5—C6—C1	119.6 (3)
O3—S1—N2	103.56 (12)	C7—C6—C1	123.1 (2)
O2—S1—C8	108.21 (12)	N1—C7—C6	124.2 (2)
O3—S1—C8	110.29 (12)	N1—C7—H7	117.9
N2—S1—C8	106.12 (10)	C6—C7—H7	117.9
C1—O1—Cu1	126.98 (15)	C9—C8—C13	122.1 (2)

C7—N1—N2	114.94 (18)	C9—C8—S1	119.4 (2)
C7—N1—Cu1	126.16 (17)	C13—C8—S1	118.46 (19)
N2—N1—Cu1	118.78 (15)	C8—C9—C10	118.1 (3)
N1—N2—S1	113.53 (15)	C8—C9—H9	121.0
N1—N2—H2N	112 (2)	C10—C9—H9	121.0
S1—N2—H2N	108 (2)	C11—C10—C9	120.7 (3)
O1—C1—C2	119.2 (2)	C11—C10—H10	119.7
O1—C1—C6	123.1 (2)	C9—C10—H10	119.7
C2—C1—C6	117.7 (2)	C12—C11—C10	120.6 (3)
C3—C2—C1	121.3 (3)	C12—C11—H11	119.7
C3—C2—H2	119.4	C10—C11—H11	119.7
C1—C2—H2	119.4	C11—C12—C13	119.5 (3)
C4—C3—C2	120.0 (3)	C11—C12—H12	120.2
C4—C3—H3	120.0	C13—C12—H12	120.2
C2—C3—H3	120.0	C12—C13—C8	119.1 (3)
C5—C4—C3	120.6 (3)	C12—C13—H13	120.5
C5—C4—Cl1	113.5 (2)	C8—C13—H13	120.5
N1^{i}—Cu1—O1—C1	155.72 (18)	O1—C1—C6—C5	−177.2 (2)
N1—Cu1—O1—C1	−24.28 (18)	C2—C1—C6—C5	2.3 (3)
O1^{i}—Cu1—N1—C7	−159.40 (19)	O1—C1—C6—C7	6.2 (3)
O1—Cu1—N1—C7	20.60 (19)	C2—C1—C6—C7	−174.3 (2)
O1^{i}—Cu1—N1—N2	16.33 (15)	N2—N1—C7—C6	176.1 (2)
O1—Cu1—N1—N2	−163.67 (15)	Cu1—N1—C7—C6	−8.1 (3)
C7—N1—N2—S1	−85.6 (2)	C5—C6—C7—N1	173.5 (2)
Cu1—N1—N2—S1	98.25 (16)	C1—C6—C7—N1	−9.8 (4)
O2—S1—N2—N1	65.37 (18)	O2—S1—C8—C9	−10.6 (2)
O3—S1—N2—N1	−166.16 (16)	O3—S1—C8—C9	−144.6 (2)
C8—S1—N2—N1	−49.98 (19)	N2—S1—C8—C9	103.9 (2)
Cu1—O1—C1—C2	−164.03 (17)	O2—S1—C8—C13	171.64 (18)
Cu1—O1—C1—C6	15.5 (3)	O3—S1—C8—C13	37.7 (2)
O1—C1—C2—C3	176.4 (2)	N2—S1—C8—C13	−73.9 (2)
C6—C1—C2—C3	−3.1 (4)	C13—C8—C9—C10	0.2 (4)
C1—C2—C3—C4	1.1 (4)	S1—C8—C9—C10	−177.5 (2)
C2—C3—C4—C5	1.9 (4)	C8—C9—C10—C11	0.2 (5)
C2—C3—C4—Cl1	−177.3 (2)	C9—C10—C11—C12	−0.5 (5)
C3—C4—C5—C6	−2.6 (4)	C10—C11—C12—C13	0.5 (4)
Cl1—C4—C5—C6	176.62 (19)	C11—C12—C13—C8	−0.1 (4)
C4—C5—C6—C7	177.3 (2)	C9—C8—C13—C12	−0.3 (4)
C4—C5—C6—C1	0.5 (4)	S1—C8—C13—C12	177.43 (18)

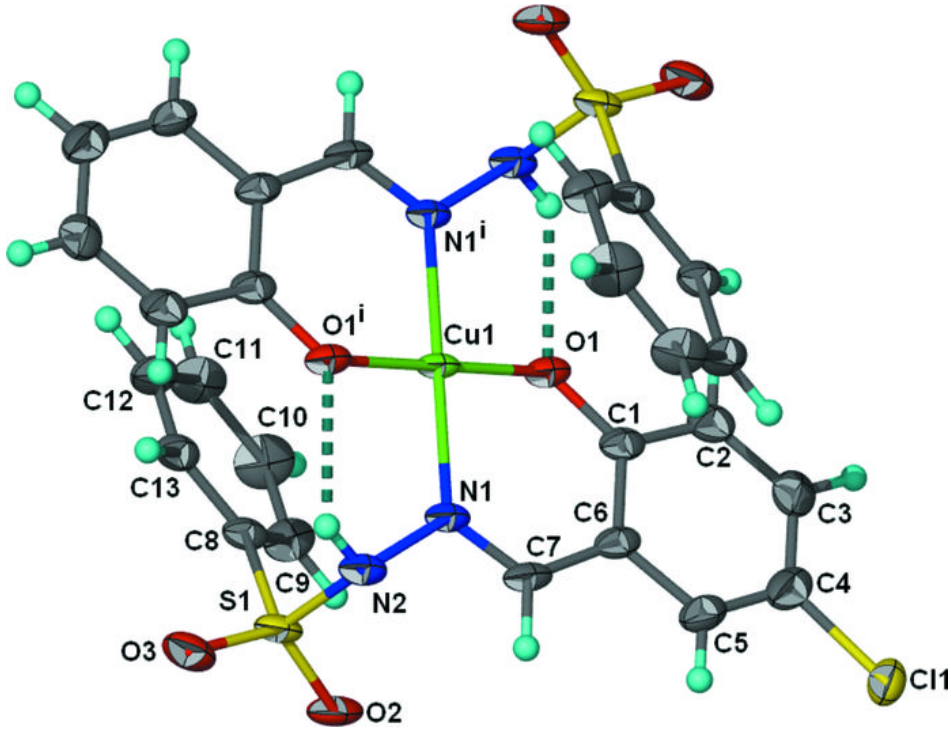
Symmetry codes: (i) −*x*+1, −*y*+1, −*z*+1.

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N2—H2N···O1^{i}	0.87 (3)	2.13 (3)	2.723 (2)	125 (3)

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

Fig. 1


N1i
O1i
Cu1
O1
C11
C12
C10
C1
C2
C13
N1
C6
C3
C8
C7
C9
S1
N2
C4
O3
C5
Cl1
O2