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Bis[*N,N*-diisobutyl-*N'*-(2-thienylcarbonyl)-thioureato]copper(II)

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The title complex, [Cu(C₁₄H₂₁N₂OS₂)₂], is isostructural with the nickel analogue, forming a square-planar coordination environment. The Cu atom lies on a crystallographic twofold rotation axis.

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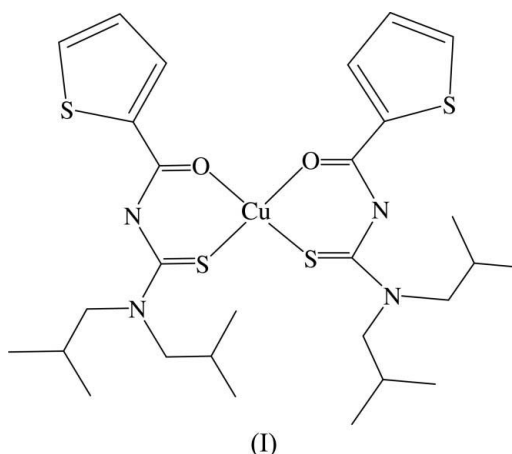
Comment

The isostructural complex bis[*N,N*-diisobutyl-*N'*-(2-thienylcarbonyl)thioureato]nickel(II) is reported in the preceding paper (Gomes, Santos, Schröder, Wagner & Low, 2007). The title complex, (I), has a square-planar geometry with an S₂O₂ coordination environment, in which the ligands assume a *cis* arrangement in relation to each other. The Cu atom lies on a crystallographic twofold rotation axis.

Key indicators

Single-crystal X-ray study
T = 290 K
Mean σ (C—C) = 0.006 Å
Disorder in main residue
R factor = 0.029
wR factor = 0.072
Data-to-parameter ratio = 17.0

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



The molecular structure of (I) is shown in Figs. 1 and 2. The main bond lengths are given in Table 1. When compared with the values for the free ligand (Gomes, Santos, Schröder & Wagner, 2007) (listed second in the following sequence of paired values), the N1—C21 [1.331 (4)/1.383 (3) Å] and N1—C6 [1.337 (4)/1.421 (3) Å] bonds are shorter, while C6—S2 [1.731 (3)/1.677 (2) Å] and C21—O2 [1.267 (3)/1.218 (3) Å], involving the coordinating O and S atoms, are longer. These observations suggest that the electrons of the C6—N1—C5 system are more delocalized in the complex.

In the crystal structure, the molecule at (*x*, *y*, *z*) is hydrogen bonded *via* a C3—H3···O2 hydrogen bond and its symmetry-related bonds, generated by the $\bar{4}$ inversion at ($\frac{1}{2}$, $\frac{1}{2}$, 0), to the molecule at (*y*, 1 − *x*, 2 − *z*), forming a hydrogen-bonded *R*₄²(20) ring (Bernstein *et al.*, 1995) (Table 2 and Fig. 3).

Experimental

The preparation of the complex was carried out according to the general procedure of Beyer *et al.* (1975). The purity of the synthesized

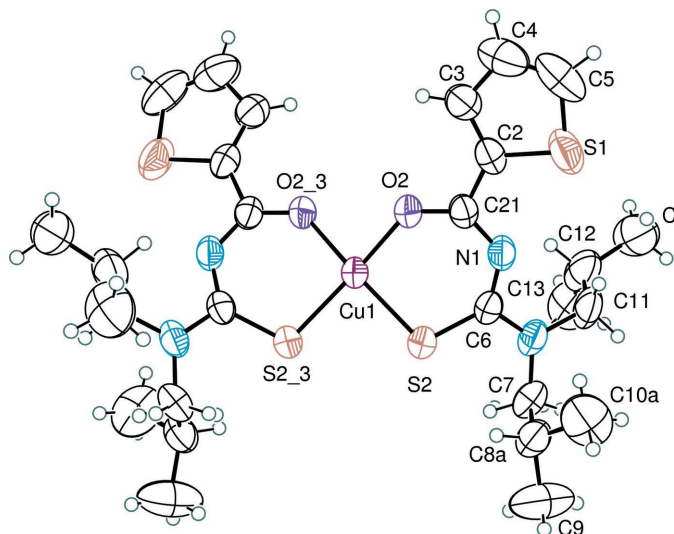


Figure 1
The molecular structure of (I), showing disorder component A and the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: $(_3) 1 - x, 1 - y, -z$.]

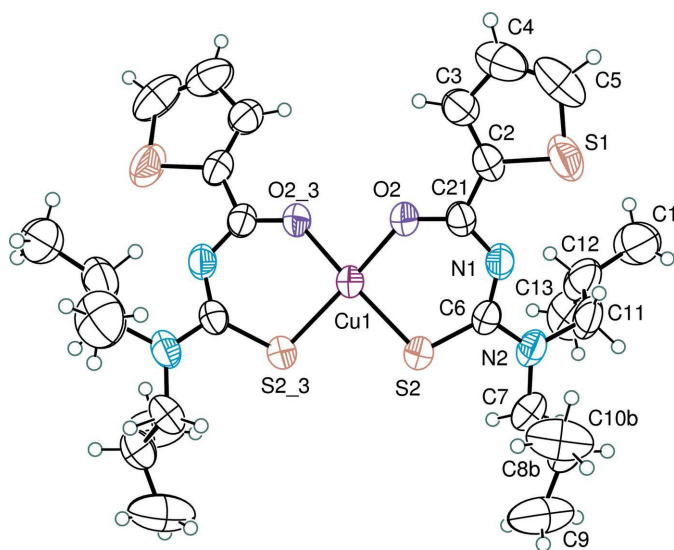


Figure 2
The molecular structure of (I), showing disorder component B and the atom-labelling scheme. Displacement ellipsoids are drawn at the 30% probability level. [Symmetry code: $(_3) 1 - x, 1 - y, -z$.]

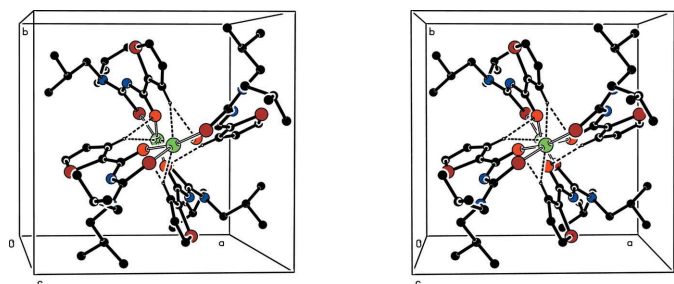


Figure 3
Stereoscopic view of the hydrogen-bonded complex of (I). H atoms not involved in the hydrogen bonding have been omitted for clarity. Only one component of disorder is shown. Hydrogen bonds are shown as dashed lines.

compounds was verified by elemental analysis. Found (%w) for $C_{28}H_{42}CuO_2S_4$: C 51.4, H 6.2, N 8.7, O 5.1, S = 20.0; calculated (%w): C 51.08, H 6.43, N 8.51, O 4.86, S 19.48.

Crystal data

$[Cu(C_{14}H_{21}N_2OS_2)_2]$
 $M_r = 658.44$
Tetragonal, $I\bar{4}$
 $a = 13.1693 (6) \text{ \AA}$
 $c = 20.2499 (16) \text{ \AA}$
 $V = 3512.0 (4) \text{ \AA}^3$

$Z = 4$
Mo $K\alpha$ radiation
 $\mu = 0.89 \text{ mm}^{-1}$
 $T = 290 (2) \text{ K}$
 $0.3 \times 0.1 \times 0.1 \text{ mm}$

Data collection

Stoe Stadi-4 diffractometer
Absorption correction: ψ scan
(North *et al.*, 1968)
 $T_{\min} = 0.804$, $T_{\max} = 0.915$
3447 measured reflections
3133 independent reflections

2938 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.016$
12 standard reflections
frequency: 60 min
intensity decay: none

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.073$
 $S = 1.14$
3133 reflections
184 parameters
H-atom parameters constrained

$\Delta\rho_{\max} = 0.26 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.17 \text{ e \AA}^{-3}$
Absolute structure: Flack (1983),
1665 Friedel pairs
Flack parameter: 0.007 (13)

Table 1

Selected bond lengths (\AA).

Cu1—O2	1.9295 (18)	N1—C6	1.337 (4)
Cu1—S2	2.2343 (7)	C6—N2	1.336 (3)
C21—O2	1.267 (3)	C6—S2	1.731 (3)
C21—N1	1.331 (4)	N2—C7	1.464 (4)

Table 2

Hydrogen-bond geometry ($\text{\AA}, ^\circ$).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C3—H3 \cdots O2 ⁱ	0.93	2.38	3.264 (6)	158

Symmetry code: (i) $y, -x + 1, -z + 2$.

H atoms bonded to C atoms were refined with standard distances (0.93 \AA for aromatic and 0.98, 0.96 and 0.97 \AA , for tertiary, secondary and primary aliphatic groups, respectively), with $U_{\text{iso}}(\text{H}) = 1.5U_{\text{eq}}(\text{C})$ for methyl groups and $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ for all others. Atoms C8 and C10 of the isobutyl group are disordered over two sites with refined occupancies of 0.496 (12) and 0.504 (12). The H atoms on C7, C8, C9 and C10 were calculated on the basis of this disorder. Those for methyl atoms C9 and C10 were calculated as six equally spaced half-occupancy H atoms. The C—C bond distances within the disordered isobutyl group were restrained to 1.520 (5) \AA . There is a solvent-accessible void in the structure of volume 51 \AA^3 , centred at the origin, and at $(\frac{1}{2}, \frac{1}{2}, \frac{1}{2})$, corresponding to 1.5% of the unit-cell volume. No discernible peaks could be found in the void.

Data collection: *IPDS Software* (Stoe & Cie, 1996); cell refinement: *CELL* (Buzzlaff, 1996); data reduction: *INTEGRATE* in *IPDS Software*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

metal-organic papers

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