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(3,5-Dimethyl-1*H*-pyrazole-κN^2)(4-oxidopent-3-en-2-one 2-hydroxybenzoylhydrazonato-$\kappa^2 N,O$)-copper(II)

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The Cu^{II} atom in the title complex, [Cu($C_{12}H_{12}N_2O_3$)($C_5H_8N_2$)], is coordinated by two carboxyl O atoms and one hydrazine N atom from the 4-oxidopent-3-en-2-one 2-hydroxybenzoylhydrazone ligand (sph^{2-}) and one N atom of 3,5-dimethylpyrazole, forming a distorted square-planar geometry.

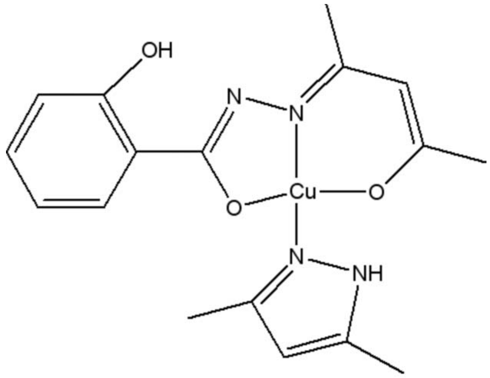
Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.004 Å
R factor = 0.033
wR factor = 0.091
Data-to-parameter ratio = 16.8

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

Comment

Transition metal complexes containing pyrazole-derived ligands have attracted much attention from chemists and biologists because of their interesting coordination chemistry and biological activities (Álvarez-Boo *et al.*, 2003; Barika *et al.*, 1999; Mukherjee, 2000; Trofimenko, 1993). The complexes of salicylic acid and its derivatives possess structural variety owing to their rich coordination modes. The incorporation of salicylyl hydrazone into coordination compounds gives interesting supramolecular architectures (Abuhijleh & Woods, 2001; Gibney *et al.*, 1993; Adams *et al.*, 2000). Here, we report the crystal structure of a copper(II) complex with salicylyl 4-keto-2-pentanone hydrazone and 3,5-dimethyl-pyrazole.



(I)

The Cu atom in complex (I) (Fig. 1) has a square-planar geometry; selected bond lengths and angles are given in Table 1, similar to those in related compounds (Kaizer *et al.*, 2006; Zang *et al.*, 2006; Yuan *et al.*, 2005). Three donor atoms (O2, N2 and O3) of the ligand sph^{2-} chelate the Cu atom, forming a five-membered and a six-membered ring. The fourth donor atom N3 comes from 3,5-dimethylpyrazole. The C7—N1 bond distance [1.319 (3) Å] is equivalent to the N2—C9 bond [1.313 (3) Å]. The O2—C7 bond distance [1.296 (2) Å] is longer than a usual double linkage C═O [1.213 Å; Kaizer *et al.*, 2006], and the C7—N1 bond distance is shorter than a usual C—N single bond [1.323, 1.366 Å; Bogdanović *et al.*, 2005], suggesting some delocalization of the double bond in

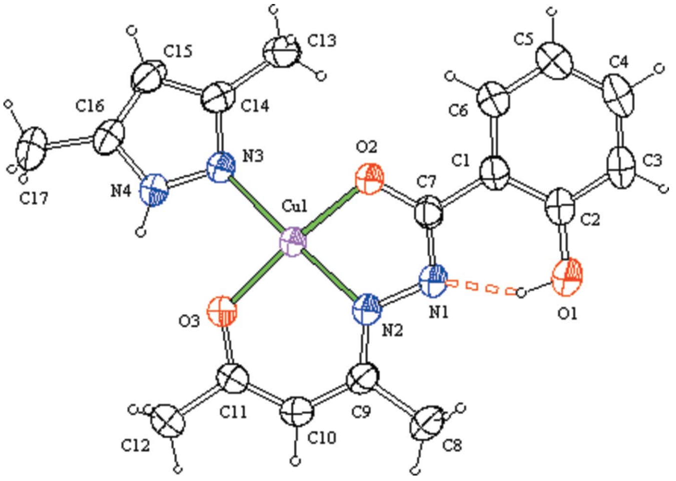


Figure 1
The molecular structure of complex (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are shown as small spheres of arbitrary radii. The broken line indicates a hydrogen bond.

the O2—C7—N1 fragment. The same is true for the C10—C11—O3 fragment. O2 and O3 are deprotonated, showing that the hydrazone ligand is in the dianionic form.

There is an intramolecular hydrogen bond, O1—H01···N1, in this compound.

Experimental

A methanol solution (20 ml) of salicylyl hydrazine (0.304 g) was dropped into a methanol solution of 2,4-pentanedione (0.2 ml) (1:1 molar ratio) with stirring and then refluxed for 2 h. The solvent was evaporated under vacuum to dryness, yielding the ligand, H_2sph, as a white powder. The ligand was recrystallized from ethyl alcohol. The ligand (23.0 mg, 0.1 mmol) and copper(II) acetate (20.0 mg, 0.1 mmol) (1:1 molar ratio) were dissolved in methanol solution (40 ml) with stirring. After stirring for half an hour, the mixture was filtered and the deep-blue solution was left to stand, allowing slow evaporation of the solvent at room temperature. Four days later, deep-blue crystals of (I) were obtained, suitable for X-ray analysis; these were washed with diethyl ether.

Crystal data

$[Cu(C_{12}H_{12}N_2O_3)(C_5H_8N_2)]$
$M_r = 391.91$
Triclinic, $P\bar{1}$
$a = 8.0713$ (7) Å
$b = 10.6660$ (11) Å
$c = 10.9429$ (8) Å
$\alpha = 110.393$ (4)°
$\beta = 96.174$ (3)°
$\gamma = 96.299$ (4)°

$V = 866.87$ (13) $Å^3$
$Z = 2$
$D_x = 1.501$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 1.28$ mm^{-1}
$T = 293$ (2) K
Block, deep-blue
0.62 × 0.53 × 0.45 mm

Data collection

Rigaku Weissenberg IP area-detector diffractometer
ω scans
Absorption correction: ψ scan (*TEXRAY*; Molecular Structure Corporation, 1999)

$T_{min} = 0.538$, $T_{max} = 0.774$ (expected range = 0.390–0.561)
8035 measured reflections
3907 independent reflections
3549 reflections with $I > 2\sigma(I)$
$R_{int} = 0.035$
$\theta_{max} = 27.5°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.091$
$S = 1.07$
3907 reflections
232 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.047P)^2 + 0.356P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.32$ e $Å^{-3}$
$\Delta\rho_{min} = -0.49$ e $Å^{-3}$

Table 1
Selected geometric parameters (Å, °).

Cu1—O3	1.9153 (14)	Cu1—O2	1.9361 (14)
Cu1—N2	1.9180 (16)	Cu1—N3	1.9911 (16)
O3—Cu1—N2	93.22 (6)	O3—Cu1—N3	89.24 (6)
O3—Cu1—O2	173.97 (6)	N2—Cu1—N3	173.60 (7)
N2—Cu1—O2	81.76 (6)	O2—Cu1—N3	96.10 (6)

The H atom of O1 was located in a difference map and then refined as riding in the as-found relative position; the U_{iso} value was refined freely. The other H atoms were placed in calculated positions and treated using a riding model with C—H = 0.93 or 0.96 Å and N—H = 0.86 Å; $U_{iso}(H) = 1.2U_{eq}(C,N)$ or $1.5U_{eq}(C)$ for H atoms in the rings and methyl H atoms, respectively.

Data collection: *TEXRAY* (Molecular Structure Corporation, 1999); cell refinement: *TEXRAY*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEX* (McArdle, 1995); software used to prepare material for publication: *SHELXL97*/2.

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