(Benzoylacetonato)chloro[3-(dimethylamino)propylamine]copper(II)

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In the molecular structure of the title complex, [Cu(C$_{10}$H$_9$O$_2$)Cl(C$_5$H$_{14}$N$_2$)], the Cu^{II} atom is in a distorted square-pyramidal geometry, coordinated by two O atoms of a benzoylacetonate ligand and two N atoms of a 3-(dimethylamino)propylamine ligand in the basal plane, while a Cl atom occupies the apical position.

Comment

In recent years, the study of the metal coordination of β-diketonate and its derivatives has attracted great interest. The metal-organic chemical vapour deposition (MOCVD) method is an established technology for the preparation of thin metallic films and metal complexes of β-diketonate derivatives can be good MOCVD precursors (Nakamori *et al.*, 1988; Donald *et al.*, 1992; Xie *et al.*, 2000). Many complexes of β-diketonate derivatives with the formula M(N—N)(O—O) (M = Cu; N—N = *o*-phenanthroline or tetramethylene-diamine; O—O = salicylaldehydate, acetylacetonate or benzoylacetonate) have been synthesized and characterized (Elmali *et al.*, 1993, 1995, 1997; Elerman *et al.*, 1995, 2000; Elerman & Geselle, 1997; Kabak *et al.*, 1999; Jalilehavand *et al.*, 1996). In the present paper the crystal structure of a related Cu^{II} complex, (I), is reported.
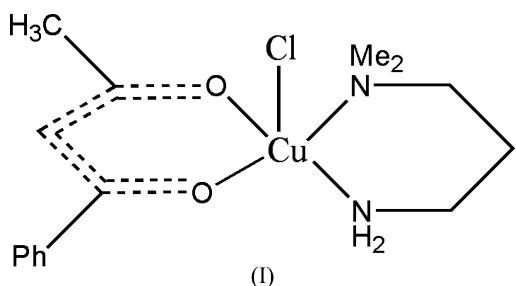
The crystal structure of (I) consists of discrete molecules (Fig. 1). The Cu^{II} atom is in a distorted square-pyramidal geometry, being coordinated by two O atoms of a benzoyl-acetonate ligand and two N atoms of a 3-dimethylamino-1-propylamine ligand in the basal plane, while a Cl atom occupies the apical position. The structure of (I) is similar to those of the previously reported complexes [CuCl(C$_{10}$H$_9$O$_2$)-(C$_{12}$H$_8$N$_2$)] (Elerman *et al.*, 2000) and [CuCl(C$_5$H$_7$O$_2$)-(C$_6$H$_{16}$N$_2$)] (Jalilehavand *et al.*, 1996). The Cu—O distances (Table 1) are comparable to those in the complex [CuCl-(C$_{10}$H$_9$O$_2$)(C$_{12}$H$_8$N$_2$)] (Elerman *et al.*, 2000), but are shorter than those observed in another complex with a mean value of 1.986 (5) Å (Heldal & Sletten, 1997). The Cu—N distances in (I) are similar to those reported for [CuCl-
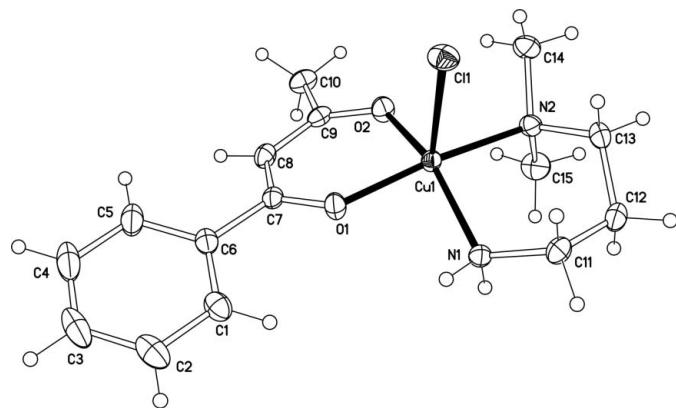
Figure 1
A view of the molecular structure of (I), showing displacement ellipsoids drawn at the 30% probability level. H atoms are shown as spheres of arbitrary radii.
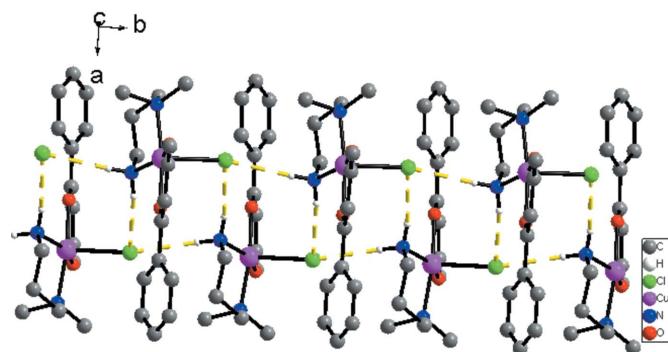

Figure 2
One-dimensional chains of the title complex. Dashed lines indicate the hydrogen bonds. H atoms not involved in hydrogen bonding have been omitted.

$(C_5H_7O_2)(C_6H_{16}N_2)$] (Jalilehavand *et al.*, 1996). The Cu—Cl bond length is in the middle of the range of the following values: 2.485 (1) (Elerman *et al.*, 2000), 2.500 (2) (Jalilehavand *et al.*, 1996), 2.615 (2) (Jian *et al.*, 2004) and 2.734 (4) Å (Phelps *et al.*, 1976). The six-membered chelate ring Cu1/N1/C11–C13/N2 adopts a chair conformation. The dihedral angles between the plane N1/C11/C13/N2 and planes C11–C13 and N1/N2/Cu1 are 58.57 (2) and 38.43 (2)°, respectively. Atoms O1/C7–C9/O2 are essentially coplanar (with r.m.s deviation 0.016 Å) and the dihedral angle between this plane and the phenyl ring is 22.70 (1)°. In the crystal structure, intermolecular N—H···Cl hydrogen bonds link molecules into one-dimensional chains along [010] (Table 2 and Fig. 2).

Experimental

A 10 ml ethanol solution of benzoylacetone (0.16 g, 1.0 mmol) was added to a stirred solution of $CuCl_2 \cdot 2H_2O$ (0.17 g, 1.0 mmol) in ethanol (10 ml). To the above mixture, 3-dimethylamino-1-propylamine (0.10 g, 1.0 mmol) was added dropwise with refluxing for about 1 h. The solution was left to stand undisturbed at room temperature for several days to yield green single crystals suitable for X-ray diffraction analysis.

Crystal data
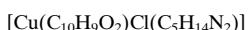
[Cu(C$_{10}$H$_9$O$_2$)Cl(C$_5$H$_{14}$N$_2$)]	$D_x = 1.494$ Mg m^{-3}
$M_r = 362.34$	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 4775
$a = 11.951$ (2) Å	reflections
$b = 6.8459$ (14) Å	$\theta = 3.2$–27.7°
$c = 20.489$ (4) Å	$\mu = 1.53$ mm^{-1}
$\beta = 106.12$ (3)°	$T = 293$ (2) K
$V = 1610.4$ (6) Å^3	Prism, green
$Z = 4$	$0.40 \times 0.32 \times 0.20$ mm

Data collection

Rigaku Saturn70 CCD (4 × 4 bin mode) diffractometer	3743 independent reflections
	3290 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.029$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	$\theta_{max} = 27.7°$
	$h = -15 \rightarrow 14$
$T_{min} = 0.562$, $T_{max} = 0.737$	$k = -8 \rightarrow 8$
12294 measured reflections	$l = -26 \rightarrow 26$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0546P)^2$
$R[F^2 > 2\sigma(F^2)] = 0.032$	$+ 0.0981P]$
$wR(F^2) = 0.087$	where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.09$	$(\Delta/\sigma)_{max} = 0.001$
3743 reflections	$\Delta\rho_{max} = 0.44$ e Å^{-3}
193 parameters	$\Delta\rho_{min} = -0.81$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.9250 (14)	O1—C7	1.270 (2)
Cu1—O2	1.9329 (13)	O2—C9	1.258 (2)
Cu1—N1	1.9877 (14)	C6—C7	1.478 (3)
Cu1—N2	2.0729 (15)	C7—C8	1.371 (3)
Cu1—Cl1	2.5812 (7)	C8—C9	1.384 (3)
O1—Cu1—O2	91.35 (6)	N2—Cu1—Cl1	91.73 (5)
O1—Cu1—N1	84.99 (6)	C7—O1—Cu1	127.16 (13)
O2—Cu1—N1	152.17 (6)	C9—O2—Cu1	126.51 (13)
O1—Cu1—N2	171.99 (6)	C11—N1—Cu1	118.41 (11)
O2—Cu1—N2	87.44 (6)	O1—C7—C6	114.61 (17)
N1—Cu1—N2	92.39 (6)	C8—C7—C6	120.60 (17)
O1—Cu1—Cl1	96.22 (4)	C7—C8—C9	124.45 (17)
O2—Cu1—Cl1	105.42 (4)	C11—C12—C13	114.47 (17)
N1—Cu1—Cl1	102.40 (5)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1B···Cl1^i	0.90	2.59	3.3974 (17)	150
N1—H1C···Cl1^{ii}	0.90	2.55	3.4379 (16)	170

Symmetry codes: (i) $-x, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $x, y - 1, z$.

All H atoms were included in calculated positions. For methyl C atoms C—H = 0.96 Å and U_{iso}(H) = 1.5U_{eq}(C) and the methyl groups were allowed to rotate to fit the electron density; for other H atoms C—H$_{aromatic}$ = 0.93 Å, C—H$_{aliphatic}$ = 0.97 Å, N—H = 0.90 Å and U_{iso}(H) = 1.2U_{eq}(C,N).

Data collection: *CrystalClear* (Rigaku/MSC, 2005); cell refinement: *CrystalClear*; data reduction: *CrystalClear*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine

structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 2002); software used to prepare material for publication: *SHELXTL*.

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