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(Benzoylacetonato)chloro(1,10-phenanthroline)copper(II)

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The crystal structure of the title compound, chloro(1,10-phenanthroline-*N*,*N*′)(1-phenyl-1,3-butanedionato-*O*,*O*′)copper(II), [CuCl(C$_{10}$H$_9$O$_2$)(C$_{12}$H$_8$N$_2$)], has been determined. The Cu^{II} ion displays a distorted square-pyramidal coordination, being linked to the two O atoms of the benzoylacetonate ligand and the two N atoms of the 1,10-phenanthroline ligand in the basal plane, and the Cl atom in the apical site. The Cu—N, Cu—O and Cu—Cl bond lengths are 2.043 (2)/2.025 (2), 1.914 (2)/1.941 (2) and 2.485 (1) Å, respectively.

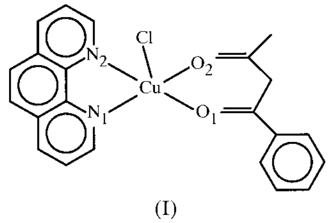
Comment

Interest in mixed-ligand chelate complexes has been clearly established in inorganic and bioinorganic chemistry in the last decade (Solans *et al.*, 1987, 1993; Moreno-Esparza *et al.*, 1995; Heldal & Sletten, 1997; Gasque *et al.*, 1999; Venkatraman *et al.*, 1999). A series of compounds of formula *M*(N–N)(O–O) (*M* = Cu; N–N = *o*-phenanthroline; O–O = salicylaldehydate, acetylacetonate or benzoylacetonate) have been synthesized and characterized (Solans *et al.*, 1987; Keramidas *et al.*, 1994). The title compound is a member of this series of complexes and its crystal structure was determined in order to obtain the ligand arrangement around the Cu^{II} ion.

We have reported previously the structures of several dimeric and monomeric Cu^{II} complexes (Elmali *et al.*, 1993, 1995, 1997; Elerman *et al.*, 1995; Elerman & Geselle, 1997; Kabak *et al.*, 1999). We report here the crystal structure of (benzoylacetonato)chloro(1,10-phenanthroline)copper(II), (I).

The Cu^{II} ion displays a distorted square-pyramidal coordination, being linked to the two O atoms of the benzoylacetonato ligand and the two N atoms of the 1,10-phenanthroline ligand in the basal plane, and the Cl atom in the apical site (Fig. 1).

The Cu—N(phenanthroline) distances [2.025 (2)–2.043 (2) Å] are similar, within experimental error, to those reported in several related complexes with mean values of 2.019 (7) (Solans *et al.*, 1988), 2.019 (2) (Moreno-Esparza *et al.*, 1995) and 2.027 (4) Å (Venkatraman *et al.*, 1999). While they are shorter than those observed in other complexes, *i.e.* mean values 2.050 (1) (Boys *et al.*, 1981) and 2.099 (4) Å (Heldal & Sletten, 1997), they are also larger than other reported values, *i.e.* mean values 2.006 (4) (Fabretti *et al.*, 1985), 1.998 (3) (Solans *et al.*, 1987) and 2.003 (3) Å (Alvarez-Larena *et al.*, 1995).



(I)

The Cu—O distances [1.914 (2)–1.941 (2) Å] are similar, within experimental error, to those reported in related complexes, *i.e.* mean values 1.925 (3) (Solans *et al.*, 1987) and 1.925 (1) Å (Gasque *et al.*, 1999), but they are shorter than those observed in another complex with a mean value of 1.986 (5) Å (Heldal & Sletten, 1997).

The π-bond character of the C14—O2 bond produces a lengthening of the Cu—O2 bond distance, 1.941 (2) Å, with respect to Cu—O1, 1.914 (2) Å. This effect is also observed in (1,10-phenanthroline)(salicylaldehydato)copper(II) nitrate (Solans *et al.*, 1987), where these distances are 1.952 (3) and 1.898 (3) Å, respectively. This fact explains the shortening of the Cu—N2 bond distance [2.025 (2) Å, *trans* to O1] with respect to Cu—N1 [2.043 (2) Å, *trans* to O2].

The Cl atom lies above the plane, at the apex of a slightly distorted pyramid. The Cu—Cl bond length [2.485 (1) Å] is intermediate when compared with the following values: 2.192 (1) (Chiari *et al.*, 1987), 2.233 (1) (Mégnamisi-Bélombé & Endres, 1983), 2.463 (1) (Nicholson *et al.*, 1982), 2.546 (2) (Solans *et al.*, 1988), 2.560 (1) (Thompson *et al.*, 1996) and 2.734 (4) Å (Phelps *et al.*, 1976).

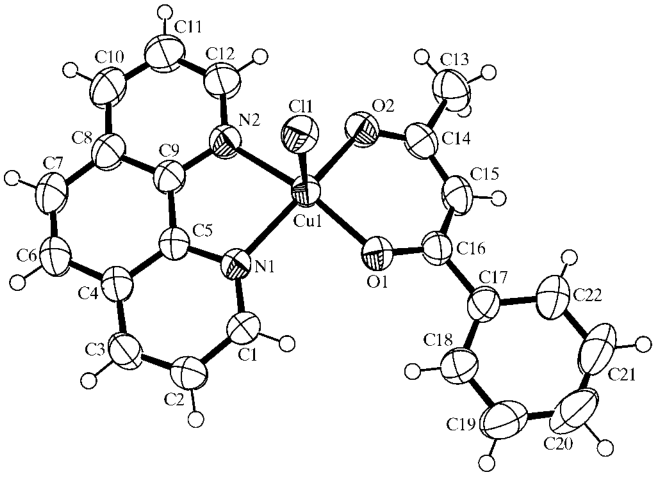


Figure 1
The molecular structure of the title compound (*ORTEP*-3; Farrugia, 1997) showing the atom-labelling scheme and 50% probability displacement ellipsoids for the non-H atoms. H atoms are drawn as spheres of arbitrary radii.

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† Alexander von Humboldt fellow.

The mean planes in the molecule are the five-membered phenanthroline chelate ring defined by atoms N1, C5, C9, N2 and Cu1, which is planar with negligible distortion (average r.m.s. deviation from the five-atom plane is 0.0199 Å), and the plane defined by atoms O1, C14, C16, O2 and Cu1, which is roughly planar (average r.m.s. deviation from the five-atom plane is 0.0246 Å). The angle between the phenanthroline chelate ring plane and the benzoylacetonate moiety excluding the C15 atom (O1, C14, C16, O2 and Cu1) is 23.37 (6)°. The angle between the planes of the coordination plane atoms and the phenanthroline molecule is 7.77 (8)°, and that formed with the benzoylacetonate moiety is 16.92 (7)°. The Cu atom is displaced by 0.28 (1) Å out of the least-squares plane containing atoms O1, O2, N1, N2 toward the apical positions.

Experimental

The complex was prepared by mixing copper(II) chloride dihydrate, 1,10-phenanthroline and benzoylacetone in a 1:1:1 ratio in ethanol. The solution was allowed to stand for several days whereupon green crystals precipitated.

Crystal data

$[CuCl(C_{10}H_9O_2)(C_{12}H_8N_2)]$
$M_r = 441.37$
Monoclinic, $C2/c$
$a = 14.453$ (3) Å
$b = 10.3993$ (13) Å
$c = 25.371$ (2) Å
$\beta = 94.3660$ (10)°
$V = 3802.2$ (9) Å^3
$Z = 8$

$D_x = 1.542$ Mg m^{-3}
Cu $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 22.56$–$42.36°$
$\mu = 3.083$ mm^{-1}
$T = 293$ (2) K
Plate, green
0.60 × 0.48 × 0.12 mm

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical *via* ψ scans (North *et al.*, 1968)
$T_{min} = 0.216$, $T_{max} = 0.691$
3860 measured reflections
3860 independent reflections

3485 reflections with $I > 2\sigma(I)$
$\theta_{max} = 74.23°$
$h = 0 \rightarrow 18$
$k = 0 \rightarrow 12$
$l = -31 \rightarrow 31$
3 standard reflections
frequency: 120 min
intensity decay: 1.36%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.053$
$wR(F^2) = 0.157$
$S = 1.064$
3860 reflections
253 parameters
H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0991P)^2 + 4.6891P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.57$ e Å^{-3}
$\Delta\rho_{min} = -0.65$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O1	1.914 (2)	Cu1—N1	2.043 (2)
Cu1—O2	1.941 (2)	Cu1—Cl1	2.4852 (9)
Cu1—N2	2.025 (2)		
O1—Cu1—O2	93.77 (9)	N2—Cu1—N1	81.09 (9)
O1—Cu1—N2	166.60 (11)	O1—Cu1—Cl1	98.99 (7)
O2—Cu1—N2	90.19 (10)	O2—Cu1—Cl1	100.66 (7)
O1—Cu1—N1	90.67 (9)	N2—Cu1—Cl1	92.84 (9)
O2—Cu1—N1	158.21 (10)	N1—Cu1—Cl1	99.69 (7)
N1—C5—C9—N2	0.5 (4)	C15—C16—C17—C22	18.1 (5)
O2—C14—C15—C16	−1.7 (5)	O1—C16—C17—C18	14.3 (4)
C14—C15—C16—O1	4.5 (5)		

Data collection: *CAD*-4 *Software* (Enraf–Nonius, 1993); cell refinement: *CAD*-4 *Software*; data reduction: *REDU*4 (Stoe & Cie, 1991); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*-3 (Farrugia, 1997); software used to prepare material for publication: *SHELXL*97.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: GS1092). Services for accessing these data are described at the back of the journal.

References

Alvarez-Larena, A., Briansó-Penalva, J. L. & Piniella, J. F. (1995). *Acta Cryst.* C**51**, 852–854.
Boys, D., Escobar, C. & Martínez-Carrera, S. (1981). *Acta Cryst.* B**37**, 351–355.
Chiari, B., Piovesana, O., Tarantelli, T. & Zanazzi, P. F. (1987). *Inorg. Chem.* **26**, 952–955.
Elerman, Y., Atakol, O., Svoboda, I. & Geselle, M. (1995). *Acta Cryst.* C**51**, 1520–1522.
Elerman, Y. & Geselle, M. (1997). *Acta Cryst.* C**53**, 549–551.
Elmali, A., Atakol, O., Svoboda, I. & Fuess, H. (1993). *Z. Kristallogr.* **203**, 271–274.
Elmali, A., Atakol, O., Svoboda, I. & Fuess, H. (1995). *Acta Cryst.* C**51**, 2520–2522.
Elmali, A., Elerman, Y., Svoboda, I., Fuess, H., Griesar, K. & Haase, W. (1997). *Z. Naturforsch. Teil B*, **52**, 157–161.
Enraf–Nonius (1993). *CAD*-4 *Software*. Release 5.1. Enraf–Nonius, Delft, The Netherlands.
Fabretti, A. C., Franchini, G., Zannini, P. & Divaira, M. (1985). *Inorg. Chim. Acta*, **105**, 187–191.
Farrugia, L. J. (1997). *J. Appl. Cryst.* **30**, 565.
Gasque, L., Moreno-Esparza, R., Molins, E., Briansó-Penalva, J. L., Ruiz-Ramírez, L. & Medina-Dickinson, G. (1999). *Acta Cryst.* C**55**, 158–160.
Heldal, H. E. & Sletten, J. (1997). *Acta Chem. Scand.* **51**, 122–124.
Kabak, M., Elmali, A., Kavlakoglu, E., Elerman, Y. & Durlu, T. N. (1999). *Acta Cryst.* C**55**, 1650–1652.
Keramidas, K. G., Voutsas, G. P., Rentzeperis, P. I. & Tsiamis, C. (1994). *Z. Kristallogr.* **209**, 52–55.
Mégnamisi-Bélombé, M. & Endres, H. (1983). *Acta Cryst.* C**39**, 707–709.
Moreno-Esparza, R., Molins, E., Briansó-Penalva, J. L., Ruiz-Ramírez, L. & Redón, R. (1995). *Acta Cryst.* C**51**, 1505–1508.
Nicholson, G. A., Petersen, J. L. & McCormick, B. J. (1982). *Inorg. Chem.* **21**, 3274–3278.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.
Phelps, D. W., Goodman, W. H. & Hodgson, D. J. (1976). *Inorg. Chem.* **15**, 2266–2271.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Solans, X., Ruiz-Ramírez, L., Gasque, L. & Briansó, J. L. (1987). *Acta Cryst.* C**43**, 428–430.
Solans, X., Ruiz-Ramírez, L., Martinez, A., Gasque, L. & Briansó, J. L. (1988). *Acta Cryst.* C**44**, 628–631.
Solans, X., Ruiz-Ramírez, L., Martinez, A., Gasque, L. & Moreno-Esparza, R. (1993). *Acta Cryst.* C**49**, 890–893.
Stoe & Cie (1991). *REDU*4. Version 7.08. Stoe & Cie, Darmstadt, Germany.
Thompson, L. K., Mandal, S. K., Tandon, S. S., Bridson, J. N. & Park, M. K. (1996). *Inorg. Chem.* **35**, 3117–3125.
Venkatraman, R., Zubkowski, J. D. & Valente, E. J. (1999). *Acta Cryst.* C**55**, 1241–1243.

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