Acta Crystallographica Section E
Structure Reports
Online

ISSN 1600-5368

μ-Oxalato-1κ^2*O*,*O*′:2κ^2*O*″,*O*‴-bis(chloro{[1-(2-pyridyl-κ*N*)ethylidene]hydrazine-κ*N*}copper(II))

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Received 7 December 2006
Accepted 2 January 2007

Key indicators

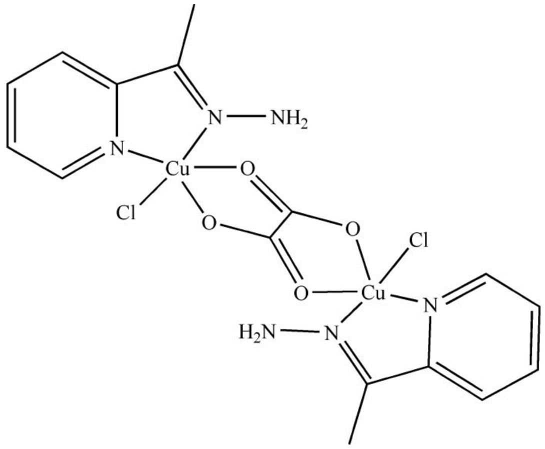
Single-crystal X-ray study
T = 100 K
Mean σ(C–C) = 0.004 Å
R factor = 0.037
wR factor = 0.099
Data-to-parameter ratio = 15.7

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

The title compound, $[Cu_2(C_2O_4)Cl_2(C_7H_9N_3)_2]$, contains neutral molecules of a centrosymmetric dinuclear oxalate-bridged copper(II) complex, in which the oxalate ligand is coordinated in a bis-bidentate bridging mode to the Cu atoms. Each Cu atom has a distorted square-pyramidal environment, being coordinated by two N atoms of the [1-(pyridin-2-yl)ethylidene]hydrazine ligand, two O atoms of the doubly deprotonated oxalate anion and one Cl^- anion.

Comment

It is well known that derivatives of oxamide are widely used in molecular magnetism for the assembly of homo- and heterometallic polynuclear complexes (Kahn, 1987; Ojima & Nonoyama, 1988; Cador *et al.*, 1997; Ruiz *et al.*, 1993). One of the strategies for obtaining polynuclear clusters is the reaction of mononuclear complexes with salts of metals or with coordinatively unsaturated complexes. However, when oxamide or its derivatives are used, hydrolysis of the primary amide groups often occurs. The title compound, (I), incorporates the products of the alkaline hydrolysis of bis{2′-[1-(pyridin-2-yl)ethylidene]oxalohydrazide} in the presence of Cu^{II} ions. Such products are the oxalate anion and [1-(pyridin-2-yl)ethylidene]hydrazine (*L*).



(I)

In (I) (Fig. 1), the Cu atom has a square-pyramidal environment, with one O atom of the oxalate bridge (O1), two N atoms of the pyridine ring and the azomethine group of *L*, and the Cl^- anion, occupying the base of the pyramid, and the second O atom of the oxalate bridge (O2) in the apical position. The Cu atom is 0.2282 (3) Å above the pyramid base. The ligand *L* is planar and forms a dihedral angle of 11.20 (7)° with the metallic basal coordination plane. The planar μ_4-oxalate

doi:10.1107/S1600536807000098

group lies on a centre of symmetry and bridges the two Cu atoms in a bis(chelating) mode, each Cu atom being bound to two O atoms from the two different carboxylic acid groups. The shortest Cu$\cdots$Cu distance is 5.4489 (11) Å.

In the crystal packing of (I), the dimers are connected to each other through hydrogen bonds, where Cl^- and oxalate O1 atoms act as acceptors and NH_2 groups act as donors (Fig. 2, Table 1).

Experimental

$CuCl_2\cdot 2H_2O$ (0.171 g, 1 mmol) was dissolved in water (15 ml) and added to an aqueous solution (15 ml) of bis{2′-[1-(pyridin-2-yl)ethylidene]oxalohydrazide} (0.324 g, 1 mmol). A few drops of HCl (1 *M* solution) were then added to the dark-green suspension. The mixture was stirred for 30 min at ambient temperature. The resulting solution was filtered and left at room temperature for crystallization in air. Bright-green crystals of (I) were separated by filtration after 72 h, washed with cold water (10 ml) and air-dried.

Crystal data

$[Cu_2(C_2O_4)Cl_2(C_7H_9N_3)_2]$
$M_r = 556.34$
Monoclinic, $P2_1/n$
$a = 9.3785$ (6) Å
$b = 9.2002$ (6) Å
$c = 11.2501$ (8) Å
$\beta = 100.422$ (6)°
$V = 954.6$ (1) Å^3
$Z = 2$
$D_x = 1.936$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 2.55$ mm^{-1}
$T = 100$ (2) K
Block, bright green
0.23 × 0.17 × 0.11 mm

Data collection

Nonius KappaCCD area-detector diffractometer
φ scans, and ω scans with κ offset
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
$T_{min} = 0.597$, $T_{max} = 0.757$
6391 measured reflections
2241 independent reflections
1988 reflections with $I > 2\sigma(I)$
$R_{int} = 0.074$
$\theta_{max} = 28.4°$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.037$
$wR(F^2) = 0.099$
$S = 1.09$
2241 reflections
143 parameters
H atoms treated by a mixture of independent and constrained refinement
$w = 1/[\sigma^2(F_o^2) + (0.0537P)^2 + 0.2578P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.79$ e Å^{-3}
$\Delta\rho_{min} = -1.09$ e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N3—H1$N\cdots$Cl1^i	0.90 (3)	2.63 (3)	3.330 (2)	135 (3)
N3—H2$N\cdots$O1^i	0.88 (3)	2.29 (3)	3.091 (3)	152 (3)

Symmetry code: (i) $-x - \frac{1}{2}, y - \frac{1}{2}, -z + \frac{3}{2}$.

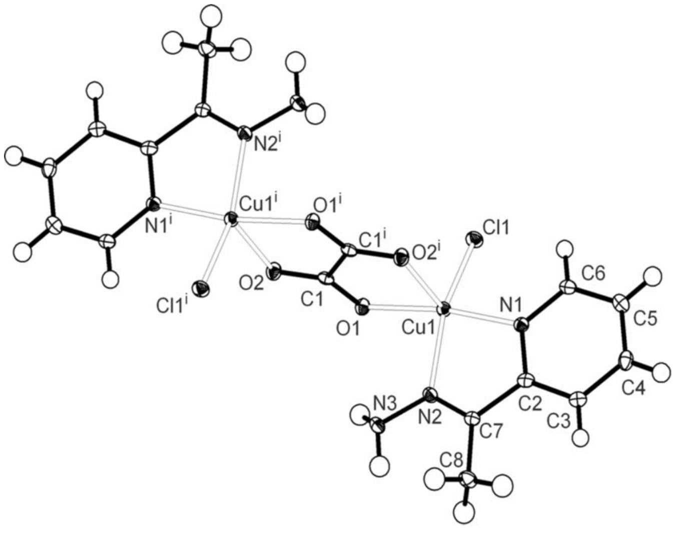


Figure 1
The molecular structure of compound (I), with displacement ellipsoids drawn at the 50% probability level and H atoms shown as spheres of arbitrary radius. [Symmetry code: (i) $-x, -y, 2 - z$.]

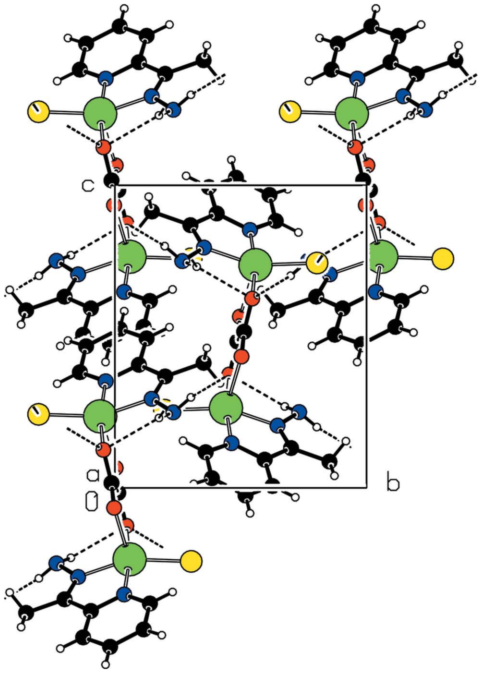


Figure 2
A packing diagram of the title compound. Hydrogen bonds are indicated by dashed lines. H atoms not involved in hydrogen bonding have been omitted for clarity.

All H atoms were observed in a difference Fourier map, but the C-bound H atoms of the methyl groups and pyridine ring were positioned geometrically and treated using a riding model, with C—H = 0.96 Å and $U_{iso}(H) = 1.2U_{eq}(C)$ for the aromatic H atoms, and with C—H = 0.93 Å and $U_{iso}(H) = 1.5U_{eq}(C)$ for the methyl H atoms. The coordinates of the N-bound H atoms were refined freely, with $U_{iso}(H) = 1.5U_{eq}(N)$. The deepest hole in the final difference Fourier map lies within 1 Å of atom Cu1.

Data collection: *KM-4 CCD Software* (Kuma Diffraction, 1999); cell refinement: *SAINT* (Bruker, 1999); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *DIAMOND* (Brandenburg, 2006); software used to prepare material for publication: *WinGX* (Farrugia, 1999).

The authors thank NATO for financial support (grant No. CBP.NUKR.CLG 982019).

References

Brandenburg, K. (2006). *DIAMOND*. Release 3.1b. Crystal Impact GbR, Bonn, Germany.
Bruker (1999). *SAINT*. Version 6.02. Bruker AXS Inc., Madison, Wisconsin, USA.
Cador, O., Mathonier, C. & Kahn, O. (1997). *Inorg. Chem.* **36**, 1923–1928.
Farrugia, L. J. (1999). *J. Appl. Cryst.* **32**, 837–838.
Kahn, O. (1987). *Struct. Bond.* **68**, 89–167.
Kuma Diffraction (1999). *KM-4 CCD Software.* Version 1.61. Kuma Diffraction, Wrocław, Poland.
Ojima, H. & Nonoyama, K. (1988). *Coord. Chem. Rev.* **92**, 85–111.
Ruiz, R., Sanz, J., Cervera, B., Lloret, F., Julve, M., Bois, C., Faus, J. & Muñoz, M. C. (1993). *J. Chem. Soc. Dalton Trans.* pp. 1623–1628.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Sheldrick, G. M. (2003). *SADABS*. Version 2.10. University of Göttingen, Germany.