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An oxalato-bridged copper(II) complex

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In the coordination complex μ-oxalato-bis[(isocyanato-*N*)(tetramethylethylenediamine)copper(II)], $[Cu_2(NCO)_2(C_2O_4)(C_6H_{16}N_2)_2]$, the Cu^{II} ions are five-coordinated. One Cu^{II} ion bridges to another centrosymmetry-related Cu^{II} ion through $C_2O_4^{2-}$, forming a plane with an r.m.s. deviation of 0.059 Å. The O—Cu—O angle is 79.33 (8)° and the Cu···Cu separation is 5.14 Å.

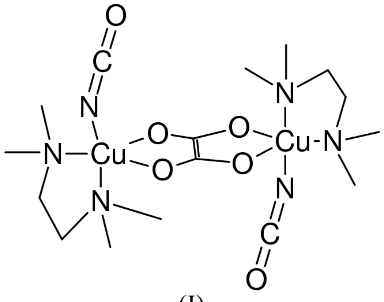
Key indicators

Single-crystal X-ray study
$T = 296$ K
Mean σ(C–C) = 0.004 Å
R factor = 0.039
wR factor = 0.090
Data-to-parameter ratio = 20.1

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

Comment

Magnetic studies of oxalate-bridged metal complexes are well documented (Julve, Verdaguer *et al.*, 1984; Julve, Faus *et al.*, 1984; Kahn, 1985). Presently, this study is mainly concentrated on one-dimensional/two-dimensional systems having alternating bridging ligands, *i.e.* systems with more than one bridging ligand (Vicente *et al.*, 1996). Recently, Ribas *et al.* (1998) designed a strategy for having Ni^{II} or Cu^{II} as central atom with the aim of combining oxalate and azide super-exchange pathways in the same compound. To get the desired compound, they replaced the water molecule in $[L(H_2O)M\text{-ox-}M(H_2O)L]^{2+}$ [$M = Cu^{II}$ (Vicente *et al.*, 1997), L = diamine; $M = Ni^{II}$ (Escuer *et al.*, 1994), L = diamine or triamine] with a bridging azide ligand. Using the same strategy, we tried to synthesize a μ-oxalato-μ-cyanato-dicopper(II) alternating chain. Surprisingly, due to the lesser bridging tendency of the cyanate ion compared to azide, we did not get the desired one-dimensional alternating chain but instead obtained a dinuclear copper(II) oxalate-bridged complex, (I), with a pendant cyanate ligand in the fifth position of each copper(II) in a *trans* fashion.



(I)

The coordination geometry about each Cu atom is distorted trigonal–bipyrimidal, with N2, N3, O2 and Cu atoms in the equatorial plane of the bipyramid (r.m.s. deviation 0.019 Å); N1 and O1 are on the axial sites. The μ-oxalato chelate is shared by both of the centrosymmetry-related Cu^{II} ions. Several similiar oxalato-briged structures of Cu^{II} coordination

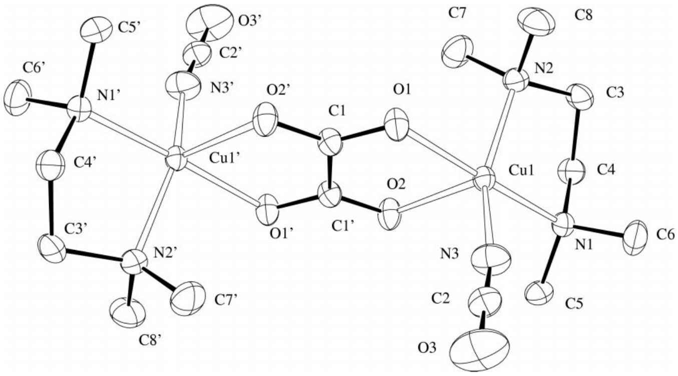


Figure 1

ORTEP drawing of the title complex. Atoms labeled with a prime are centrosymmetrically related to those without prime. The coordination bonds are hollow.

complexes with aqua instead of isocyanato have been reported (Julve, Verdaguer *et al.*, 1984; Julve, Faus *et al.*, 1984; Sletten, 1983).

Experimental

N,N,N′,N′-Tetramethylethane-1,2-diamine (0.3 ml, 2 mmol) was added slowly to copper nitrate trihydrate (483.2 mg, 2 mmol) dissolved in water (10 ml). To the deep-blue solution, an aqueous solution (10 ml) of potassium cyanate (162.6 mg, 2 mmol) was added drop-by-drop with constant stirring and a blue crystalline compound separated out. An aqueous solution (10 ml) of sodium oxalate (134 mg, 1 mmol) was added slowly with vigorous stirring and a deep-blue solution was obtained. This was filtered and the filtrate was kept in a desiccator. After a few days, single crystals suitable for X-ray crystal structure analysis were obtained.

Crystal data

$[Cu_2(NCO)_2(C_2O_4)(C_6H_{16}N_2)_2]$
$M_r = 531.06$
Monoclinic, $P2_1/c$
$a = 7.4943$ (9) Å
$b = 14.5660$ (17) Å
$c = 10.8812$ (13) Å
$\beta = 105.655$ (2)°
$V = 1143.8$ (2) Å^3
$Z = 2$
$D_x = 1.543$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
$\theta = 2.4$–28.3°
$\mu = 1.90$ mm^{-1}
$T = 296$ (2) K
Rectangular plate, blue
0.23 × 0.10 × 0.10 mm

Data collection

CCD area-detector diffractometer
φ and ω scans
Absorption correction: empirical (North *et al.*, 1968)
$T_{\min} = 0.646$, $T_{\max} = 0.827$
7265 measured reflections
2734 independent reflections
2367 reflections with $I > 2\sigma(I)$
$R_{\text{int}} = 0.032$
$\theta_{\max} = 28.3°$
$h = -9 \rightarrow 9$
$k = -15 \rightarrow 18$
$l = -14 \rightarrow 14$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.039$
$wR(F^2) = 0.090$
$S = 1.16$
2734 reflections
136 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.115P)^2 + 0.612P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max} = 0.37$ e Å^{-3}
$\Delta\rho_{\min} = -0.51$ e Å^{-3}

Table 1

Selected geometric parameters (Å, °).

Cu1—N3	1.933 (3)	O1—C1^i	1.261 (3)
Cu1—O1	1.9946 (19)	C1—O2	1.247 (3)
Cu1—N1	2.030 (2)	C1—O1^i	1.261 (3)
Cu1—N2	2.062 (2)	C1—C1^i	1.540 (5)
Cu1—O2	2.2330 (19)		
N3—Cu1—O1	92.33 (11)	N1—Cu1—N2	85.91 (9)
N3—Cu1—N1	93.99 (11)	N3—Cu1—O2	103.58 (11)
O1—Cu1—N1	173.19 (9)	O1—Cu1—O2	79.23 (7)
N3—Cu1—N2	152.15 (12)	N1—Cu1—O2	96.78 (8)
O1—Cu1—N2	89.69 (9)	N2—Cu1—O2	104.09 (9)

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

Data collection: *SMART* (Bruker, 1998); cell refinement: *SMART*; data reduction: *SHELXTL* (Bruker, 1998); program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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m264 Jack Cheng *et al.* • $[Cu_2(NCO)_2(C_2O_4)(C_6H_{16}N_2)_2]$ *Acta Cryst.* (2001). E**57**, m263–m264