Acta Crystallographica Section C
Crystal Structure Communications
ISSN 0108-2701

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Electronic paper
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Four copper(II) complexes with potentially tetradentate tripodal ligands

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Received 4 April 2000
Accepted 18 April 2000

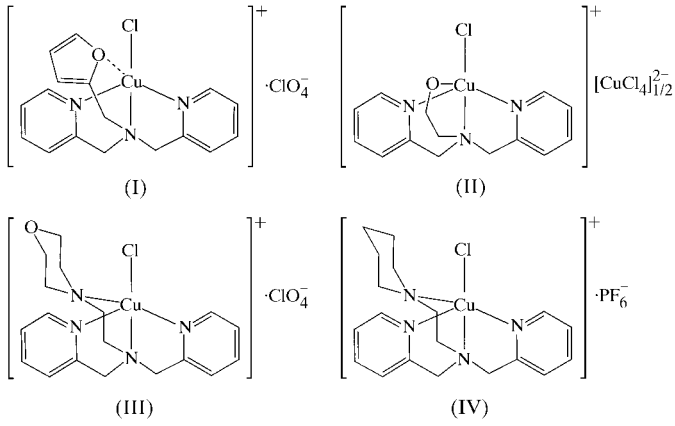
Data validation number: IUC0000116

The four title Cu^{II} compounds are chloro[(2-furylmethyl)-bis(2-pyridylmethyl)amine-*N*,*N*′,*N*″]copper(II) perchlorate, $[CuCl(C_{17}H_{17}N_3O)]ClO_4$, (I), chloro{2-[bis(2-pyridylmethyl)-amino]ethanolato-*N*,*N*′,*N*″,*O*}copper(II) hemi[tetrachloro-copper(II)], $[CuCl(C_{14}H_{17}N_3O)][CuCl_4]_{1/2}$, (II), chloro[(2-morpholinoethyl)bis(2-pyridylmethyl)amine-*N*,*N*′,*N*″,*N*‴]-copper(II) perchlorate, $[CuCl(C_{18}H_{24}N_4O)]ClO_4$, (III), and chloro[(2-piperidinylethyl)bis(2-pyridylmethyl)amine-*N*,*N*′,-*N*″,*N*‴]copper(II) hexafluorophosphate, $[CuCl(C_{19}H_{26}N_4)]$-PF_6, (IV). They have tripodal potentially tetradentate ligands. In (I), the O atom of the furan moiety weakly coordinates to the Cu atom at a distance of 2.750 (3) Å.

Comment

Relaxation of supercoiled plasmid DNA by the mononuclear copper(II) complex and hydrogen peroxide system was reported by Kobayashi *et al.* (1996). The structures of the four title Cu^{II} compounds $[Cu(fpy)Cl]ClO_4$, (I), $[Cu(etpa)Cl]$-$[CuCl_4]_{1/2}$, (II), $[Cu(mopy)Cl]ClO_4$, (III) and $[Cu(pipy)Cl]$-PF_6, (IV), are reported here.

In (I), two independent complex cations show essentially the same structure. The O atom of the furan moiety weakly coordinates to the Cu atom. The Cl ligand of a neighboring unit, related by an inversion centre, approaches the *trans* position to form a dimeric structure.



Experimental

Synthesis of $[Cu(fpy)Cl]ClO_4$, (I): to a stirred solution of $CuCl_2{\cdot}2H_2O$ (1 mmol) in methanol (50 ml) was added a methanol solution of the ligand fpy (1 mmol). The resulting dark-blue solution was stirred for 90 min after which$NaClO_4$ (500 mg) in methanol (10 ml) was added. The blue precipitate which formed immediately was collected and recrystallized from aqueous methanol. Crystals of $[Cu(etpa)Cl]$-$[CuCl_4]_{1/2}$, (II), $[Cu(mopy)Cl]ClO_4$, (III), and $[Cu(pipy)Cl]PF_6$, (IV), were prepared by a similar method.

Compound (I)

Crystal data

$[CuCl(C_{17}H_{17}N_3O)]ClO_4$	$Z = 4$
$M_r = 477.79$	$D_x = 1.637$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 13.658$ (2) Å	Cell parameters from 25 reflections
$b = 14.199$ (3) Å	$\theta = 10$–$15°$
$c = 11.720$ (3) Å	$\mu = 1.437$ mm^{-1}
$\alpha = 104.92$ (2)°	$T = 299$ K
$\beta = 112.08$ (2)°	Plate, blue
$\gamma = 68.32$ (1)°	0.5 × 0.4 × 0.2 mm
$V = 1938.5$ (8) Å^3	

Data collection

Rigaku AFC-5*S* diffractometer	$R_{int} = 0.011$
θ–2θ scans	$\theta_{max} = 27.5°$
Absorption correction: by integration (Coppens *et al.*, 1965)	$h = 0 \rightarrow 18$
$T_{min} = 0.477$, $T_{max} = 0.780$	$k = -18 \rightarrow 18$
9268 measured reflections	$l = -15 \rightarrow 15$
8895 independent reflections	3 standard reflections
6332 reflections with $I > 2\sigma(I)$	every 100 reflections
	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters not refined
$R(F) = 0.046$	$w = 1/[\sigma^2(F_o^2) + \{0.1(F_o^2 + 2F_c^2)/3\}^2]$
$wR(F^2) = 0.148$	$(\Delta/\sigma)_{max} = 0.003$
$S = 0.99$	$\Delta\rho_{max} = 0.42$ e Å^{-3}
8895 reflections	$\Delta\rho_{min} = -0.53$ e Å^{-3}
505 parameters	

Table 1
Selected geometric parameters (Å, °) for (I).

Cu1—Cl1	2.248 (1)	Cu1—N2	2.034 (3)
Cu1—Cl1^{i}	2.933 (1)	Cu1—N3	1.984 (3)
Cu1—O1	2.750 (3)	Cu2—Cl2	2.244 (1)
Cu1—N1	1.977 (3)	Cu2—Cl2^{ii}	2.896 (1)

Cu2—O2	2.864 (4)	Cu2—N5	2.046 (3)
Cu2—N4	1.977 (3)	Cu2—N6	1.985 (3)
O1—Cu1—Cl1^{i}	160.22 (6)	Cu1—Cl1^{i}—Cu1^{i}	85.85 (3)
O2—Cu2—Cl2^{ii}	165.23 (7)	Cu2—Cl2^{ii}—Cu2^{ii}	85.58 (3)

Symmetry codes: (i) $1-x, 1-y, 1-z$; (ii) $2-x, 2-y, 1-z$.

Compound (II)

Crystal data

$[Cu(C_{14}H_{17}N_3O)Cl][CuCl_4]_{1/2}$	D_x = 1.720 Mg m^{-3}
M_r = 444.99	Mo $K\alpha$ radiation
Monoclinic, $C2/c$	Cell parameters from 25 reflections
a = 23.377 (4) Å	θ = 10–15°
b = 9.204 (2) Å	μ = 2.343 mm^{-1}
c = 16.361 (3) Å	T = 295 K
β = 102.43 (1)°	Plate, dark blue
V = 3437.8 (10) Å^3	0.6 × 0.5 × 0.4 mm
Z = 8	

Data collection

Rigaku AFC-5*S* diffractometer	R_{int} = 0.041
θ–2θ scans	θ_{max} = 27.5°
Absorption correction: by integration (Coppens *et al.*, 1965)	$h = 0 \rightarrow 30$
T_{min} = 0.331, T_{max} = 0.446	$k = 0 \rightarrow 12$
4290 measured reflections	$l = -21 \rightarrow 21$
3943 independent reflections	3 standard reflections every 100 reflections
3182 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters not refined
$R(F)$ = 0.040	$w = 1/[\sigma^2(F_o^2) + \{0.1(F_o^2 + 2F_c^2)/3\}^2]$
$wR(F^2)$ = 0.131	$(\Delta/\sigma)_{max}$ = 0.026
S = 1.00	$\Delta\rho_{max}$ = 0.51 e Å^{-3}
3943 reflections	$\Delta\rho_{min}$ = −0.86 e Å^{-3}
207 parameters	

Table 2
Selected geometric parameters (Å) for (II).

Cu1—Cl1	2.244 (1)	Cu1—N2	2.050 (2)
Cu1—O1	2.306 (2)	Cu1—N3	2.001 (3)
Cu1—N1	1.987 (3)		

Compound (III)

Crystal data

$[CuCl(C_{18}H_{24}N_4O)]ClO_4$	D_x = 1.599 Mg m^{-3}
M_r = 510.86	Mo $K\alpha$ radiation
Monoclinic, $P2_1/n$	Cell parameters from 25 reflections
a = 9.282 (3) Å	θ = 10–15°
b = 11.592 (4) Å	μ = 1.319 mm^{-1}
c = 19.731 (3) Å	T = 299 K
β = 90.76 (2)°	Prism, blue
V = 2122.8 (9) Å^3	0.6 × 0.3 × 0.2 mm
Z = 4	

Data collection

Rigaku AFC-5*S* diffractometer	R_{int} = 0.015
θ–2θ scans	θ_{max} = 27.5°
Absorption correction: by integration (Coppens *et al.*, 1965)	$h = 0 \rightarrow 12$
T_{min} = 0.638, T_{max} = 0.799	$k = 0 \rightarrow 15$
5433 measured reflections	$l = -26 \rightarrow 26$
4878 independent reflections	3 standard reflections every 100 reflections
3479 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters not refined
$R(F)$ = 0.049	$w = 1/[\sigma^2(F_o^2) + \{0.1(F_o^2 + 2F_c^2)/3\}^2]$
$wR(F^2)$ = 0.197	$(\Delta/\sigma)_{max}$ = 0.011
S = 1.32	$\Delta\rho_{max}$ = 0.59 e Å^{-3}
4878 reflections	$\Delta\rho_{min}$ = −0.48 e Å^{-3}
271 parameters	

Table 3
Selected geometric parameters (Å) for (III).

Cu1—Cl1	2.243 (1)	Cu1—N3	2.000 (4)
Cu1—N1	1.993 (4)	Cu1—N4	2.399 (4)
Cu1—N2	2.059 (4)		

Compound (IV)

Crystal data

$[CuCl(C_{19}H_{26}N_4)]PF_6$	D_x = 1.599 Mg m^{-3}
M_r = 554.41	Mo $K\alpha$ radiation
Monoclinic, $P2_1/c$	Cell parameters from 25 reflections
a = 11.855 (3) Å	θ = 10–15°
b = 15.258 (2) Å	μ = 1.197 mm^{-1}
c = 13.186 (3) Å	T = 299 K
β = 105.14 (2)°	Prism, blue
V = 2302.2 (7) Å^3	0.6 × 0.3 × 0.3 mm
Z = 4	

Data collection

Rigaku AFC-5*S* diffractometer	R_{int} = 0.026
θ–2θ scans	θ_{max} = 25.0°
Absorption correction: by integration (Coppens *et al.*, 1965)	$h = 0 \rightarrow 14$
T_{min} = 0.654, T_{max} = 0.717	$k = 0 \rightarrow 18$
4439 measured reflections	$l = -15 \rightarrow 15$
4048 independent reflections	3 standard reflections every 100 reflections
2903 reflections with $I > 2\sigma(I)$	intensity decay: none

Refinement

Refinement on F^2	H-atom parameters not refined
$R(F)$ = 0.045	$w = 1/[\sigma^2(F_o^2) + \{0.1(F_o^2 + 2F_c^2)/3\}^2]$
$wR(F^2)$ = 0.147	$(\Delta/\sigma)_{max}$ = 0.002
S = 0.99	$\Delta\rho_{max}$ = 0.33 e Å^{-3}
4048 reflections	$\Delta\rho_{min}$ = −0.27 e Å^{-3}
289 parameters	

Table 4
Selected geometric parameters (Å) for (IV).

Cu1—Cl1	2.243 (1)	Cu1—N3	1.998 (3)
Cu1—N1	2.006 (3)	Cu1—N4	2.428 (3)
Cu1—N2	2.056 (3)		

The positional parameters of all the H atoms were calculated geometrically and fixed with U(H) = 1.2U_{eq}(parent atom).

For compounds (I)–(IV), data collection: *MSC/AFC Diffractometer Control Software* (Molecular Structure Corporation, 1993); cell refinement: *MSC/AFC Diffractometer Control Software*; data reduction: *TEXSAN* (Molecular Structure Corporation, 1999); program(s) used to refine structure: *TEXSAN*; software used to prepare material for publication: *TEXSAN*. For compounds (I), (III) and (IV), program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1994). For compound (II), program(s) used to solve structure: *DIRDIF*94 (Beurskens *et al.*, 1994).

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