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cis-[*N*-(4-Chlorobenzyl)iminodiacetato-κ^3N,O,O']bis(1*H*-imidazole-κN^3)-copper(II)

María Pilar Brandi-Blanco,[a] María Magdalena de Benavides-Giménez,[a] Josefa María González-Pérez[a] and Duane Choquesillo-Lazarte[b]*

[a]Departamento de Química Inorgánica, Facultad de Farmacia, Universidad de Granada, c/ Campus de Cartuja s/n, 18071 Granada, Spain, and [b]Laboratorio de Estudios Cristalográficos, IACT – CSIC, P. T. Ciencias de la Salud, Granada 18100, Spain
Correspondence e-mail: duanec@ugr.es

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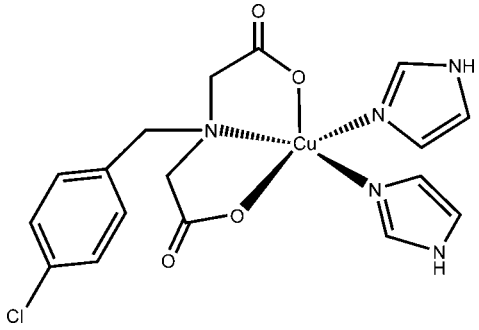
Key indicators: single-crystal X-ray study; T = 298 K; mean σ(C–C) = 0.003 Å; R factor = 0.032; wR factor = 0.086; data-to-parameter ratio = 17.5.

In the title compound, $[Cu(C_{11}H_{10}ClNO_4)(C_3H_4N)_2]$, the Cu^{II} atom is in a square-pyramidal coordination geometry, with the two imidazole ligands in *cis* positions and the *N*-(4-chlorobenzyl)iminodiacetate ligand occupying the apical and two *cis*-basal positions. In the crystal structure, molecules are linked into sheets by N—H···O hydrogen bonds.

Related literature

In addition to a related dinuclear iminodiacetate derivative (Nguyen-Huy *et al.*, 1990), it is known that a closely related *N*-(benzyl)iminodiacetate(2−) derivative exists, also as an Him solvate (Him is imidazole) (Polyakova *et al.*, 2001).

For related literature, see: Addison *et al.* (1984).



Experimental

Crystal data

$[Cu(C_{11}H_{10}ClNO_4)(C_3H_4N)_2]$
M_r = 455.35
Monoclinic, $P2_1/n$
a = 10.2028 (5) Å
b = 13.4280 (6) Å
c = 13.9113 (7) Å
β = 97.119 (1)°
V = 1891.20 (16) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 1.33 mm^{-1}
T = 298 (2) K
0.49 × 0.32 × 0.15 mm

Data collection

Bruker SMART APEX CCD area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)
T_{min} = 0.562, T_{max} = 0.825
21629 measured reflections
4437 independent reflections
4043 reflections with $I > 2\sigma(I)$
R_{int} = 0.024

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.032
$wR(F^2)$ = 0.086
S = 1.05
4437 reflections
253 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.55 e Å^{-3}
$\Delta\rho_{min}$ = −0.48 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—O8	1.9556 (13)	Cu1—N1	2.0855 (14)
Cu1—N21	1.9874 (15)	Cu1—O4	2.2252 (13)
Cu1—N31	2.0009 (15)		
O8—Cu1—N21	89.70 (6)	N31—Cu1—N1	97.54 (6)
O8—Cu1—N31	152.89 (6)	O8—Cu1—O4	108.66 (6)
N21—Cu1—N31	92.12 (6)	N21—Cu1—O4	96.01 (6)
O8—Cu1—N1	84.05 (5)	N31—Cu1—O4	98.04 (6)
N21—Cu1—N1	169.05 (6)	N1—Cu1—O4	77.60 (5)

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

D—H···A	D—H	H···A	D···A	D—H···A
N23—H23···O4^{i}	0.85	1.90	2.733 (2)	164
N33—H33···O9^{ii}	0.86	1.93	2.779 (2)	169

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

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *publCIF* (Westrip, 2007).

Financial support from ERDF-EC, MEC Spain (project CTQ2006–15329-C02–02/BQU) is gratefully acknowledged. DCh-L thanks CSIC-EU for an I3P postdoctoral research contract.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: PR2009).

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supplementary materials

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cis-[*N*-(4-Chlorobenzyl)iminodiacetato-κ^3N,O,O']bis(1*H*-imidazole-κN^3)copper(II)

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Comment

The molecular structure of (I) (Fig. 1) is centrosymmetric. The Cu(II) atom exhibits a square pyramidal coordination geometry, with a τ parameter of 0.27 (Addison *et al.*, 1984), built by a *N*-(*p*-chlorobenzyl)iminodiacetato ligand in a *fac*-tridentate conformation and two *cis* imidazole ligands. In the dianionic ligand, the two five-membered chelate rings lie nearly perpendicular (dihedral angle 72.1 (1)°).

In the crystal, centrosymmetric pairs of molecules are H-bonded by two N—H···O interactions involving parallel Him (N23) ligands which are weakly π,π-stacked (Fig. 2). Additional N—H···O interactions connect pairs of molecules generating sheets (Fig. 3).

Experimental

The new ternary complex $[Cu^{II}(C_{11}H_{10}ClNO_4)(C_3H_4N)_2]$ was obtained by reaction of a mixture having $Cu_2CO_3(OH)_2$/H_2L/Him 1 mmol/2 mmol/20 mmol (H_2L, *N*-(*p*-chlorobenzyl)iminodiacetic acid; Him, imidazole) in water (200 ml); with Him in a large excess. The resulting solution was filtered on a crystallization device which was covered with a plastic film to control the evaporation. After multiple days, prismatic blue crystals were collected and used for X-ray diffraction studies.

Refinement

The amine (imidazole) H atoms were located in the difference map and refined as riding, with N—H = 0.86 Å and with U_{iso}(H) = 1.2U_{eq}(N). Other H atoms were positioned geometrically and treated as riding with C—H = 0.95–0.99 Å, and with U_{iso}(H) = 1.2U_{eq}(C).

Figures

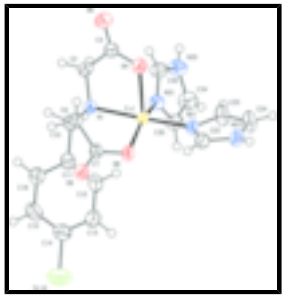

Fig. 1. A view of the asymmetric unit of (I), showing the atom-numbering scheme. Displacement ellipsoids are drawn at the 50% probability level. H atoms are represented as spheres of arbitrary radii.

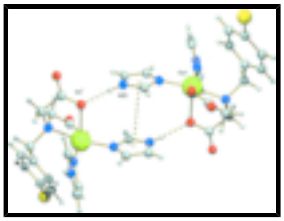

Fig. 2. A pair of centrosymmetric molecules of (I) built by N—H···O and π,π-interactions (dashed lines). Symmetry code (i): *-x, −y, −z*.

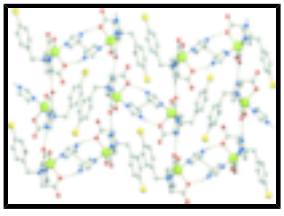

Fig. 3. A view of part of the crystal structure of (I), showing the formation of a sheet. The hydrogen bonds and π,π stacking interactions are shown as dashed lines and for the sake of clarity the H atoms bonded to C atoms have been omitted..

cis-[*N*-(4-Chlorobenzyl)iminodiacetato-κ^3*N*,*O*,*O*']bis(1*H*-imidazole- κ*N*^{3})copper(II)

Crystal data

$[Cu(C_{11}H_{10}ClNO_4)(C_3H_4N)_2]$	F_{000} = 932
M_r = 455.35	D_x = 1.599 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 9904 reflections
a = 10.2028 (5) Å	θ = 2.3–27.8º
b = 13.4280 (6) Å	μ = 1.33 mm^{-1}
c = 13.9113 (7) Å	T = 298 (2) K
β = 97.1190 (10)º	Prism, blue
V = 1891.20 (16) Å^3	0.49 × 0.32 × 0.15 mm
Z = 4	

Data collection

Bruker SMART APEX CCD area-detector diffractometer	4043 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.024
T = 298(2) K	θ_{max} = 28.3º
φ and ω scans	θ_{min} = 2.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	h = −13→13
T_{min} = 0.562, T_{max} = 0.825	k = −17→17
21629 measured reflections	l = −18→18
4437 independent reflections	

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites

$R[F^2 > 2\sigma(F^2)] = 0.032$	H-atom parameters constrained
$wR(F^2) = 0.086$	$w = 1/[\sigma^2(F_o^2) + (0.0462P)^2 + 0.8491P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.05$	$(\Delta/\sigma)_{max} = 0.001$
4437 reflections	$\Delta\rho_{max} = 0.55$ e Å^{-3}
253 parameters	$\Delta\rho_{min} = -0.47$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted *R*-factor *wR* and goodness of fit *S* are based on F^2, conventional *R*-factors *R* are based on *F*, with *F* set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating *R*-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. *R*-factors based on F^2 are statistically about twice as large as those based on *F*, and *R*- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	*y*	*z*	U_{iso}*/U_{eq}
Cu1	0.245284 (19)	0.061386 (15)	0.202469 (15)	0.02673 (8)
N1	0.42366 (14)	0.13906 (11)	0.21938 (11)	0.0286 (3)
C2	0.39633 (19)	0.24419 (13)	0.19177 (15)	0.0357 (4)
H2A	0.4771	0.2751	0.1764	0.043*
H2B	0.3680	0.2795	0.2464	0.043*
C3	0.28983 (18)	0.25385 (14)	0.10470 (14)	0.0334 (4)
O4	0.20768 (13)	0.18265 (10)	0.09352 (10)	0.0367 (3)
O5	0.28859 (16)	0.33030 (12)	0.05607 (13)	0.0534 (4)
C6	0.49925 (17)	0.09147 (13)	0.14677 (14)	0.0309 (4)
H6A	0.5931	0.0976	0.1682	0.037*
H6B	0.4806	0.1261	0.0854	0.037*
C7	0.46428 (17)	−0.01813 (13)	0.13216 (13)	0.0294 (3)
O8	0.34874 (12)	−0.04288 (9)	0.14890 (10)	0.0340 (3)
O9	0.54492 (13)	−0.07508 (10)	0.10158 (11)	0.0390 (3)
C10	0.4993 (2)	0.13779 (15)	0.31940 (14)	0.0382 (4)
H10A	0.5713	0.1852	0.3219	0.046*
H10B	0.4413	0.1591	0.3656	0.046*
C11	0.55438 (19)	0.03720 (15)	0.34877 (14)	0.0354 (4)
C12	0.4757 (2)	−0.03648 (16)	0.38096 (15)	0.0391 (4)
H12	0.3882	−0.0224	0.3882	0.047*
C13	0.5256 (2)	−0.13117 (17)	0.40257 (15)	0.0429 (5)
H13	0.4726	−0.1808	0.4240	0.052*
C14	0.6554 (2)	−0.14963 (16)	0.39155 (16)	0.0452 (5)
Cl14	0.71813 (8)	−0.26918 (5)	0.41465 (7)	0.0785 (2)

C15	0.7374 (2)	−0.0771 (2)	0.3637 (2)	0.0544 (6)
H15	0.8257	−0.0909	0.3589	0.065*
C16	0.6863 (2)	0.01688 (18)	0.34306 (17)	0.0478 (5)
H16	0.7412	0.0671	0.3251	0.057*
N21	0.08104 (14)	−0.01447 (12)	0.15999 (11)	0.0313 (3)
C22	0.06850 (19)	−0.08136 (14)	0.09012 (14)	0.0342 (4)
H22	0.1385	−0.1074	0.0612	0.041*
N23	−0.05788 (16)	−0.10680 (13)	0.06625 (12)	0.0385 (4)
H23	−0.0938	−0.1400	0.0178	0.046*
C24	−0.1310 (2)	−0.05290 (18)	0.12264 (19)	0.0507 (6)
H24	−0.2223	−0.0544	0.1215	0.061*
C25	−0.0452 (2)	0.00305 (19)	0.18050 (17)	0.0484 (5)
H25	−0.0678	0.0470	0.2274	0.058*
N31	0.16465 (15)	0.12642 (12)	0.31048 (11)	0.0333 (3)
C32	0.1159 (2)	0.21764 (15)	0.30966 (15)	0.0382 (4)
H32	0.1343	0.2672	0.2666	0.046*
N33	0.03733 (18)	0.22961 (13)	0.37816 (13)	0.0432 (4)
H33	0.0069	0.2872	0.3912	0.052*
C34	0.0349 (2)	0.14175 (17)	0.42734 (16)	0.0463 (5)
H34	−0.0118	0.1283	0.4792	0.056*
C35	0.1139 (2)	0.07826 (15)	0.38561 (15)	0.0393 (4)
H35	0.1314	0.0127	0.4045	0.047*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.02382 (12)	0.02576 (13)	0.03157 (13)	−0.00142 (7)	0.00726 (8)	−0.00333 (8)
N1	0.0274 (7)	0.0249 (7)	0.0333 (7)	−0.0021 (5)	0.0031 (6)	−0.0019 (6)
C2	0.0360 (10)	0.0241 (8)	0.0469 (10)	−0.0043 (7)	0.0046 (8)	−0.0022 (8)
C3	0.0313 (9)	0.0308 (9)	0.0398 (9)	0.0016 (7)	0.0114 (7)	0.0025 (7)
O4	0.0364 (7)	0.0343 (7)	0.0383 (7)	−0.0045 (5)	−0.0003 (5)	0.0031 (6)
O5	0.0478 (9)	0.0449 (9)	0.0683 (10)	−0.0006 (7)	0.0098 (8)	0.0252 (8)
C6	0.0265 (8)	0.0296 (9)	0.0377 (9)	−0.0038 (7)	0.0082 (7)	−0.0015 (7)
C7	0.0255 (8)	0.0314 (9)	0.0316 (8)	−0.0010 (7)	0.0049 (7)	−0.0008 (7)
O8	0.0254 (6)	0.0296 (6)	0.0486 (8)	−0.0038 (5)	0.0113 (5)	−0.0092 (5)
O9	0.0299 (7)	0.0339 (7)	0.0560 (9)	0.0008 (5)	0.0167 (6)	−0.0054 (6)
C10	0.0384 (10)	0.0348 (10)	0.0393 (10)	−0.0033 (8)	−0.0035 (8)	−0.0060 (8)
C11	0.0330 (9)	0.0404 (10)	0.0314 (9)	−0.0022 (8)	−0.0017 (7)	−0.0025 (8)
C12	0.0287 (9)	0.0492 (11)	0.0391 (10)	−0.0023 (8)	0.0026 (8)	0.0010 (9)
C13	0.0398 (11)	0.0451 (11)	0.0428 (11)	−0.0094 (9)	0.0007 (8)	0.0053 (9)
C14	0.0470 (12)	0.0425 (11)	0.0456 (11)	0.0057 (9)	0.0037 (9)	0.0062 (9)
Cl14	0.0848 (5)	0.0506 (4)	0.1031 (6)	0.0214 (3)	0.0231 (4)	0.0204 (4)
C15	0.0358 (11)	0.0629 (15)	0.0663 (15)	0.0110 (10)	0.0133 (10)	0.0200 (12)
C16	0.0341 (10)	0.0507 (13)	0.0582 (13)	−0.0046 (9)	0.0043 (9)	0.0146 (10)
N21	0.0252 (7)	0.0338 (8)	0.0356 (8)	−0.0022 (6)	0.0065 (6)	−0.0010 (6)
C22	0.0328 (9)	0.0332 (9)	0.0370 (9)	−0.0026 (7)	0.0057 (7)	−0.0010 (7)
N23	0.0357 (8)	0.0392 (9)	0.0388 (8)	−0.0086 (7)	−0.0023 (7)	0.0000 (7)
C24	0.0264 (10)	0.0654 (15)	0.0606 (14)	−0.0069 (9)	0.0062 (9)	−0.0075 (11)

C25	0.0275 (10)	0.0648 (15)	0.0548 (13)	−0.0037 (9)	0.0123 (9)	−0.0177 (11)
N31	0.0355 (8)	0.0315 (8)	0.0344 (8)	0.0009 (6)	0.0104 (6)	−0.0024 (6)
C32	0.0446 (11)	0.0333 (10)	0.0388 (10)	0.0050 (8)	0.0130 (8)	0.0007 (8)
N33	0.0490 (10)	0.0373 (9)	0.0459 (9)	0.0095 (8)	0.0169 (8)	−0.0047 (7)
C34	0.0542 (13)	0.0482 (12)	0.0403 (11)	0.0032 (10)	0.0214 (9)	−0.0007 (9)
C35	0.0469 (11)	0.0328 (10)	0.0402 (10)	0.0028 (8)	0.0138 (9)	0.0026 (8)

Geometric parameters (Å, °)

Cu1—O8	1.9556 (13)	C13—C14	1.374 (3)
Cu1—N21	1.9874 (15)	C13—H13	0.9300
Cu1—N31	2.0009 (15)	C14—C15	1.370 (3)
Cu1—N1	2.0855 (14)	C14—Cl14	1.743 (2)
Cu1—O4	2.2252 (13)	C15—C16	1.382 (3)
N1—C2	1.480 (2)	C15—H15	0.9300
N1—C6	1.489 (2)	C16—H16	0.9300
N1—C10	1.505 (2)	N21—C22	1.318 (2)
C2—C3	1.529 (3)	N21—C25	1.373 (2)
C2—H2A	0.9700	C22—N23	1.335 (2)
C2—H2B	0.9700	C22—H22	0.9300
C3—O5	1.229 (2)	N23—C24	1.357 (3)
C3—O4	1.268 (2)	N23—H23	0.8528
C6—C7	1.522 (2)	C24—C25	1.343 (3)
C6—H6A	0.9700	C24—H24	0.9300
C6—H6B	0.9700	C25—H25	0.9300
C7—O9	1.237 (2)	N31—C32	1.322 (2)
C7—O8	1.274 (2)	N31—C35	1.383 (2)
C10—C11	1.500 (3)	C32—N33	1.329 (3)
C10—H10A	0.9700	C32—H32	0.9300
C10—H10B	0.9700	N33—C34	1.366 (3)
C11—C12	1.383 (3)	N33—H33	0.8605
C11—C16	1.386 (3)	C34—C35	1.353 (3)
C12—C13	1.389 (3)	C34—H34	0.9300
C12—H12	0.9300	C35—H35	0.9300
O8—Cu1—N21	89.70 (6)	C11—C12—C13	120.84 (19)
O8—Cu1—N31	152.89 (6)	C11—C12—H12	119.6
N21—Cu1—N31	92.12 (6)	C13—C12—H12	119.6
O8—Cu1—N1	84.05 (5)	C14—C13—C12	118.28 (19)
N21—Cu1—N1	169.05 (6)	C14—C13—H13	120.9
N31—Cu1—N1	97.54 (6)	C12—C13—H13	120.9
O8—Cu1—O4	108.66 (6)	C15—C14—C13	122.3 (2)
N21—Cu1—O4	96.01 (6)	C15—C14—Cl14	118.91 (18)
N31—Cu1—O4	98.04 (6)	C13—C14—Cl14	118.83 (18)
N1—Cu1—O4	77.60 (5)	C14—C15—C16	118.7 (2)
C2—N1—C6	109.27 (14)	C14—C15—H15	120.7
C2—N1—C10	108.01 (14)	C16—C15—H15	120.7
C6—N1—C10	111.90 (14)	C15—C16—C11	120.8 (2)
C2—N1—Cu1	108.30 (11)	C15—C16—H16	119.6
C6—N1—Cu1	103.12 (10)	C11—C16—H16	119.6

C10—N1—Cu1	116.03 (11)	C22—N21—C25	105.20 (16)
N1—C2—C3	112.27 (15)	C22—N21—Cu1	124.73 (13)
N1—C2—H2A	109.2	C25—N21—Cu1	128.92 (14)
C3—C2—H2A	109.2	N21—C22—N23	111.10 (17)
N1—C2—H2B	109.2	N21—C22—H22	124.4
C3—C2—H2B	109.2	N23—C22—H22	124.4
H2A—C2—H2B	107.9	C22—N23—C24	107.64 (17)
O5—C3—O4	126.93 (19)	C22—N23—H23	129.6
O5—C3—C2	117.70 (17)	C24—N23—H23	121.6
O4—C3—C2	115.28 (16)	C25—C24—N23	106.28 (19)
C3—O4—Cu1	114.37 (12)	C25—C24—H24	126.9
N1—C6—C7	111.90 (14)	N23—C24—H24	126.9
N1—C6—H6A	109.2	C24—C25—N21	109.77 (19)
C7—C6—H6A	109.2	C24—C25—H25	125.1
N1—C6—H6B	109.2	N21—C25—H25	125.1
C7—C6—H6B	109.2	C32—N31—C35	105.33 (16)
H6A—C6—H6B	107.9	C32—N31—Cu1	125.97 (13)
O9—C7—O8	124.87 (17)	C35—N31—Cu1	126.20 (13)
O9—C7—C6	119.27 (15)	N31—C32—N33	111.45 (18)
O8—C7—C6	115.78 (15)	N31—C32—H32	124.3
C7—O8—Cu1	116.00 (11)	N33—C32—H32	124.3
C11—C10—N1	113.20 (15)	C32—N33—C34	107.75 (17)
C11—C10—H10A	108.9	C32—N33—H33	121.7
N1—C10—H10A	108.9	C34—N33—H33	129.8
C11—C10—H10B	108.9	C35—C34—N33	106.30 (18)
N1—C10—H10B	108.9	C35—C34—H34	126.9
H10A—C10—H10B	107.8	N33—C34—H34	126.9
C12—C11—C16	119.00 (19)	C34—C35—N31	109.17 (18)
C12—C11—C10	121.28 (18)	C34—C35—H35	125.4
C16—C11—C10	119.72 (19)	N31—C35—H35	125.4

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N23—H23···O4^{i}	0.85	1.90	2.733 (2)	164
N33—H33···O9^{ii}	0.86	1.93	2.779 (2)	169

Symmetry codes: (i) −*x*, −*y*, −*z*; (ii) −*x*+1/2, *y*+1/2, −*z*+1/2.

Fig. 1

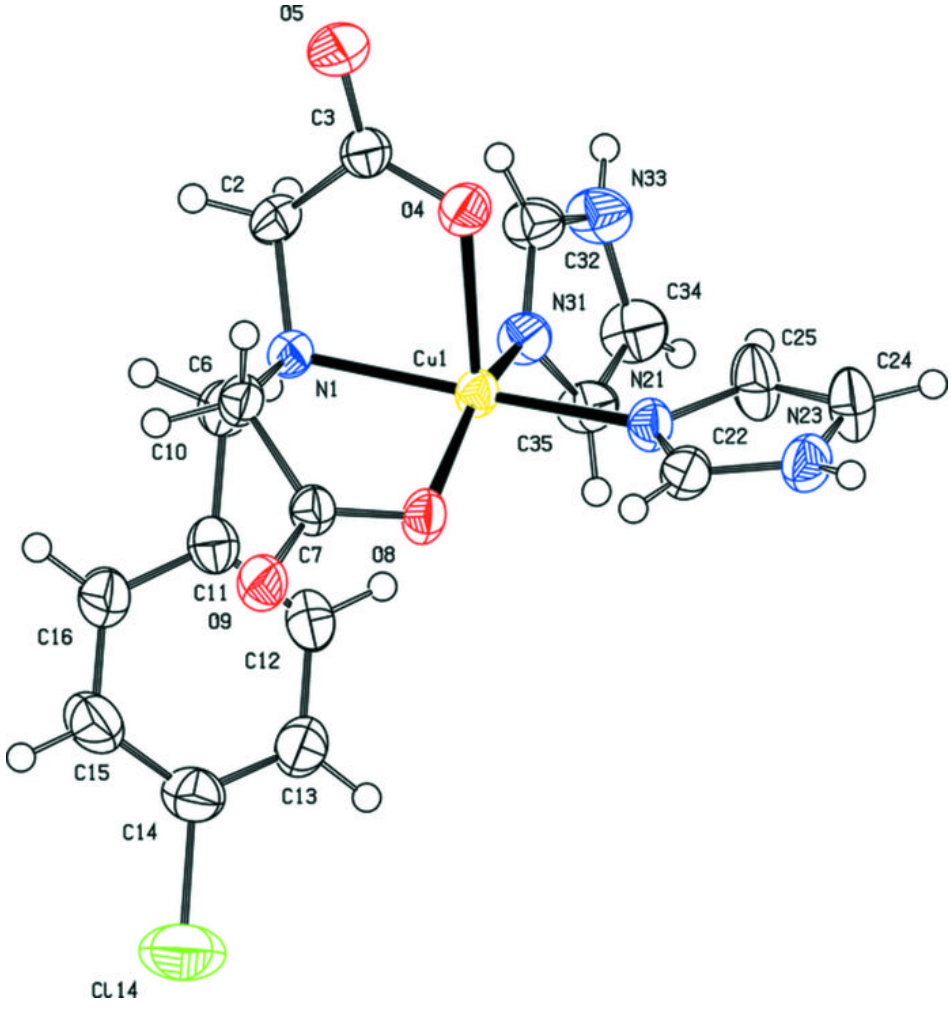
O5
C3
C2
O4
N33
C32
N31
C34
C25
C24
C6
Cu1
N1
N21
N23
C10
C35
C22
C7
O8
C11
O9
C16
C12
C15
C13
C14
CL14

Fig. 2

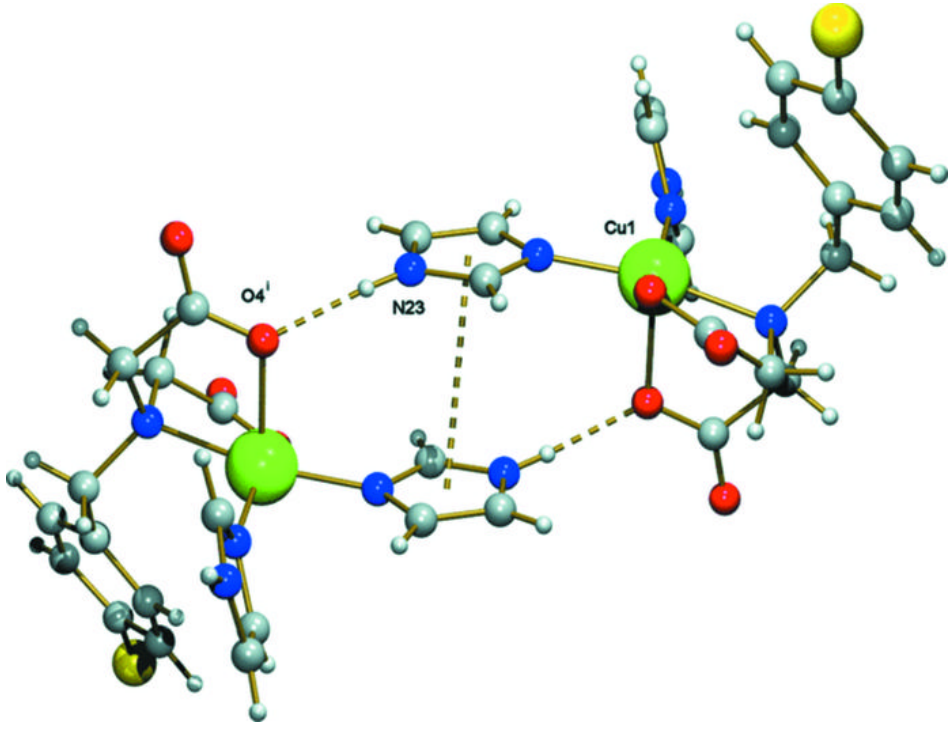

Fig. 3