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Iodidobis(1,10-phenanthroline)copper(II) perchlorate

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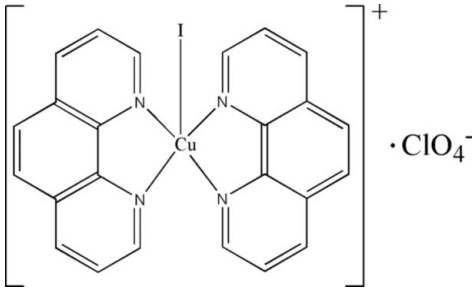
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.007 Å; R factor = 0.061; wR factor = 0.164; data-to-parameter ratio = 17.4.

The title compound, $[CuI(C_{12}H_8N_2)_2]ClO_4$, was prepared by a hydrothermal reaction at 453 K. The Cu atom has a slightly distorted trigonal–bipyramidal coordination geometry, pentacoordinated by four N atoms of two chelating 1,10-phenanthroline ligands and one I atom. There are supramolecular π–π interactions between neighbouring parallel pyridine rings, with a face-to-face distance of 3.51 (1) Å.

Related literature

For related literature, see: Bencini *et al.* (1989); Boys *et al.* (1981); Huang *et al.* (2004); Khalaji *et al.* (2007); Marta *et al.* (2006); Parker *et al.* (1994); Yu *et al.* (2004).



Experimental

Crystal data

$[CuI(C_{12}H_8N_2)_2]ClO_4$
M_r = 650.30
Monoclinic, $C2/c$
a = 16.740 (3) Å
b = 11.796 (2) Å
c = 12.558 (3) Å
β = 113.30 (3)°
V = 2277.5 (9) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 2.47 mm^{-1}
T = 295 (2) K
0.46 × 0.13 × 0.12 mm

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.370, T_{max} = 0.743
10929 measured reflections
2617 independent reflections
2296 reflections with $I > 2\sigma(I)$
R_{int} = 0.064

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.061
$wR(F^2)$ = 0.165
S = 1.02
2617 reflections
150 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 3.88 e Å^{-3}
$\Delta\rho_{min}$ = −2.86 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

I1—Cu1	2.7000 (10)	Cu1—N2	2.086 (3)
Cu1—N1	1.978 (4)		
N1^i—Cu1—N1	179.6 (2)	N2—Cu1—N2^i	118.35 (18)
N1^i—Cu1—N2	97.80 (14)	N1—Cu1—I1	90.22 (11)
N1—Cu1—N2	81.97 (14)	N2—Cu1—I1	120.82 (9)

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

Data collection: *RAPID-AUTO* (Rigaku, 2004); cell refinement: *RAPID-AUTO*; data reduction: *RAPID-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Bruker, 1997); software used to prepare material for publication: *SHELXTL*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: BX2075).

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supplementary materials

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Iodidobis(1,10-phenanthroline)copper(II) perchlorate

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Comment

In recent years, bivalent copper complexes with 1,10-phenanthroline ligand have attracted much attention for their biochemical, optical and electromagnetic applications (Marta *et al.*, 2006; Yu *et al.*, 2004; Bencini *et al.*, 1989). Many complexes have been synthesized and their structure have been reported, such as $[Cu(phen)_2Cl]ClO_4$ (Boys *et al.*, 1981) and $[Cu(phen)_2Br]ClO_4$ (Parker *et al.*, 1994) as representative examples. We report here the crystal structure of the title compound.

The structure of the title compound(I), is illustrated in Fig.1. Selected geometric parameters are listed in Table 1. The copper atom exhibits a slightly distorted trigonal-bipyramidal stereochemistry, which is five-coordinated by four nitrogen atoms from two *cis*-related chelating 1,10-phenanthroline ligands and one iodine atom.

There are supramolecular π—π interactions between neighbouring parallel pyridine rings, which help to stabilize the crystal structure of (I), and link the molecules into two-dimensional layers, with a face-to-face distance of 3.51 (1) Å (Fig. 2). The similar interactions were observed in the structures of [Ag(phen)(CN)] compound (Huang *et al.*, 2004) and $[Ag(phen)–(PPh_3)]\ ClO_4\ 0.5CH_3CN$ (Khalaji *et al.*, 2007).

Experimental

For the synthesis of complex(I), a mixture of copper(II) perchlorate hexahydrate (0.4 mmol, 146 mg), 1,10-phenanthroline (0.4 mmol, 79.2 mg), KI (0.6 mmol, 99.6 mg) and H_2O (20.0 ml) was sealed in a 40 ml stainless steel reactor with a Teflon liner and heated directly to 453 K. After maintaining this temperature for 72 h, the mixture was cooled slowly to room temperature at a rate of 3 K/h(kept at 383 K for 12 h and kept at 363 K for 72 h respectively). Black long strip crystals of the title complex were collected by filtration and were obtained in 35% yield. Analysis calculated for $C_{24}H_{16}ClCuIN_4O_4$(%): C 44.33, H 2.48, N 8.62; found: C 44.36, H 2.46, N 8.68.

Refinement

All H atoms were placed in geometrically idealized positions and were refined isotropically in the riding-model approximation. The bond lengths of C—H were fixed at 0.93 Å. The U_{iso}(H) values were set equal to 1.2 times the U_{eq} value of the C parent atoms. O atoms of the perchlorate group are slightly disordered; it was not split into two positions as its U_{eq} values are normally.

Figures

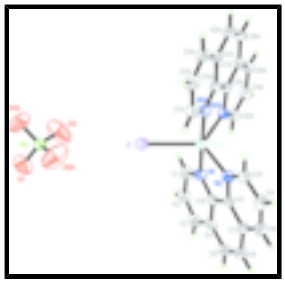

Fig. 1. The structure of complex (I), with displacement ellipsoids drawn at the 30% probability level and H atoms shown as small spheres of arbitrary radii.

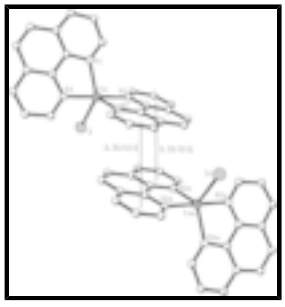

Fig. 2. Part of the crystal structure of the title compound, showing the π—π stacking interactions as dashed lines between the pyridine rings. H atoms have been omitted for clarity.

Iodidobis(1,10-phenanthroline)copper(II) perchlorate

Crystal data

$[CuI(C_{12}H_8N_2)_2]ClO_4$	F_{000} = 1276
M_r = 650.30	D_x = 1.897 Mg m^{-3}
Monoclinic, *C*2/*c*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 8209 reflections
a = 16.740 (3) Å	θ = 3.1–25.6º
b = 11.796 (2) Å	μ = 2.47 mm^{-1}
c = 12.558 (3) Å	*T* = 295 (2) K
β = 113.30 (3)º	Prism, blue
V = 2277.5 (9) Å^3	0.46 × 0.13 × 0.12 mm
Z = 4	

Data collection

Rigaku R-AXIS RAPID IP area-detector diffractometer	2617 independent reflections
Radiation source: rotating anode	2296 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.064
T = 295(2) K	θ_{max} = 27.5º
oscillation scans	θ_{min} = 3.1º
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	*h* = −21→20
T_{min} = 0.370, T_{max} = 0.743	*k* = −15→15
10929 measured reflections	*l* = −16→16

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.061$

$wR(F^2) = 0.165$

$S = 1.02$

2617 reflections

150 parameters

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0551P)^2 + 30.844P]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = 0.011$

$\Delta\rho_{max} = 3.88$ e Å^{-3}

$\Delta\rho_{min} = -2.86$ e Å^{-3}

Extinction correction: none

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds 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 $F^2 > 2$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}
I1	0.5000	0.51742 (3)	0.2500	0.05310 (10)
Cu1	0.5000	0.74631 (7)	0.2500	0.05310 (10)
Cl1	0.5000	0.12606 (12)	0.2500	0.0489 (3)
O1	0.5620 (3)	0.0559 (5)	0.2329 (5)	0.1297 (16)
O2	0.4631 (5)	0.1929 (6)	0.1527 (5)	0.174 (3)
N1	0.5935 (2)	0.7470 (3)	0.1910 (3)	0.05310 (10)
N2	0.59261 (19)	0.8369 (3)	0.3854 (3)	0.0435 (7)
C1	0.5919 (3)	0.6988 (4)	0.0937 (4)	0.0598 (12)
H1A	0.5398	0.6690	0.0410	0.072*
C2	0.6658 (3)	0.6919 (4)	0.0691 (4)	0.0637 (12)
H2A	0.6627	0.6576	0.0009	0.076*
C3	0.7419 (3)	0.7350 (4)	0.1440 (4)	0.0636 (12)
H3A	0.7918	0.7281	0.1290	0.076*
C4	0.7455 (2)	0.7909 (4)	0.2461 (4)	0.0532 (10)
C5	0.8213 (3)	0.8429 (4)	0.3292 (5)	0.0669 (13)
H5A	0.8727	0.8418	0.3175	0.080*

C6	0.8198 (3)	0.8937 (4)	0.4247 (5)	0.0638 (14)
H6A	0.8701	0.9282	0.4766	0.077*
C7	0.7429 (3)	0.8958 (4)	0.4482 (4)	0.0519 (11)
C8	0.7376 (3)	0.9452 (4)	0.5476 (4)	0.0616 (13)
H8A	0.7859	0.9803	0.6030	0.074*
C9	0.6613 (3)	0.9409 (4)	0.5616 (4)	0.0608 (12)
H9A	0.6566	0.9747	0.6258	0.073*
C10	0.5901 (3)	0.8856 (4)	0.4793 (3)	0.0516 (10)
H10A	0.5385	0.8827	0.4906	0.062*
C11	0.6678 (2)	0.8436 (3)	0.3694 (3)	0.0423 (9)
C12	0.6686 (2)	0.7934 (3)	0.2658 (3)	0.0420 (8)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
I1	0.04444 (14)	0.05585 (19)	0.06436 (17)	0.000	0.02721 (12)	0.000
Cu1	0.04444 (14)	0.05585 (19)	0.06436 (17)	0.000	0.02721 (12)	0.000
Cl1	0.0505 (6)	0.0512 (7)	0.0480 (6)	0.000	0.0226 (5)	0.000
O1	0.130 (2)	0.127 (4)	0.188 (3)	0.052 (2)	0.123 (2)	0.048 (3)
O2	0.193 (5)	0.213 (6)	0.118 (3)	0.096 (5)	0.064 (3)	0.093 (4)
N1	0.04444 (14)	0.05585 (19)	0.06436 (17)	0.000	0.02721 (12)	0.000
N2	0.0431 (12)	0.0451 (16)	0.0467 (13)	0.0017 (12)	0.0226 (11)	0.0062 (12)
C1	0.0636 (19)	0.068 (3)	0.0604 (19)	0.0010 (19)	0.0381 (15)	0.0009 (18)
C2	0.077 (2)	0.072 (3)	0.0629 (18)	0.012 (2)	0.0492 (15)	0.0108 (18)
C3	0.0593 (17)	0.071 (3)	0.080 (2)	0.0142 (18)	0.0480 (15)	0.0219 (19)
C4	0.0454 (15)	0.052 (2)	0.0714 (19)	0.0105 (15)	0.0327 (14)	0.0229 (17)
C5	0.0410 (16)	0.072 (3)	0.092 (3)	0.0059 (18)	0.0314 (18)	0.025 (2)
C6	0.0391 (17)	0.065 (3)	0.083 (3)	−0.0040 (18)	0.0193 (18)	0.012 (2)
C7	0.0446 (17)	0.046 (2)	0.059 (2)	−0.0006 (16)	0.0140 (16)	0.0133 (17)
C8	0.061 (2)	0.055 (2)	0.062 (2)	−0.009 (2)	0.0169 (19)	0.0028 (19)
C9	0.077 (2)	0.055 (2)	0.0520 (19)	−0.007 (2)	0.0283 (18)	−0.0033 (17)
C10	0.0552 (18)	0.053 (2)	0.0527 (18)	−0.0005 (16)	0.0274 (15)	0.0029 (16)
C11	0.0392 (14)	0.0412 (17)	0.0479 (16)	0.0027 (13)	0.0186 (12)	0.0122 (13)
C12	0.0396 (13)	0.0438 (18)	0.0487 (15)	0.0063 (13)	0.0240 (12)	0.0145 (13)

Geometric parameters (Å, °)

I1—Cu1	2.7000 (10)	C3—C4	1.422 (7)
Cu1—N1^{i}	1.978 (4)	C3—H3A	0.9300
Cu1—N1	1.978 (4)	C4—C12	1.404 (6)
Cu1—N2	2.086 (3)	C4—C5	1.424 (6)
Cu1—N2^{i}	2.086 (3)	C5—C6	1.349 (8)
Cl1—O2^{i}	1.378 (6)	C5—H5A	0.9300
Cl1—O2	1.378 (6)	C6—C7	1.430 (7)
Cl1—O1^{i}	1.408 (5)	C6—H6A	0.9300
Cl1—O1	1.408 (5)	C7—C11	1.398 (5)
N1—C1	1.338 (6)	C7—C8	1.411 (7)
N1—C12	1.352 (5)	C8—C9	1.357 (7)

N2—C10	1.327 (5)	C8—H8A	0.9300
N2—C11	1.354 (5)	C9—C10	1.393 (6)
C1—C2	1.392 (6)	C9—H9A	0.9300
C1—H1A	0.9300	C10—H10A	0.9300
C2—C3	1.348 (6)	C11—C12	1.434 (6)
C2—H2A	0.9300		
$N1^{i}$—Cu1—N1	179.6 (2)	C2—C3—H3A	120.1
$N1^{i}$—Cu1—N2	97.80 (14)	C4—C3—H3A	120.1
N1—Cu1—N2	81.97 (14)	C12—C4—C3	117.0 (3)
$N1^{i}$—Cu1—$N2^{i}$	81.97 (14)	C12—C4—C5	118.4 (4)
N1—Cu1—$N2^{i}$	97.80 (14)	C3—C4—C5	124.6 (4)
N2—Cu1—$N2^{i}$	118.35 (18)	C6—C5—C4	121.2 (4)
$N1^{i}$—Cu1—I1	90.22 (11)	C6—C5—H5A	119.4
N1—Cu1—I1	90.22 (11)	C4—C5—H5A	119.4
N2—Cu1—I1	120.82 (9)	C5—C6—C7	121.7 (4)
$N2^{i}$—Cu1—I1	120.82 (9)	C5—C6—H6A	119.2
$O2^{i}$—Cl1—O2	110.2 (6)	C7—C6—H6A	119.2
$O2^{i}$—Cl1—$O1^{i}$	107.5 (4)	C11—C7—C8	117.0 (4)
O2—Cl1—$O1^{i}$	111.9 (4)	C11—C7—C6	118.5 (4)
$O2^{i}$—Cl1—O1	111.9 (4)	C8—C7—C6	124.5 (4)
O2—Cl1—O1	107.5 (4)	C9—C8—C7	119.5 (4)
$O1^{i}$—Cl1—O1	108.0 (5)	C9—C8—H8A	120.3
C1—N1—C12	118.9 (4)	C7—C8—H8A	120.3
C1—N1—Cu1	127.4 (3)	C8—C9—C10	119.6 (5)
C12—N1—Cu1	113.4 (3)	C8—C9—H9A	120.2
C10—N2—C11	117.7 (3)	C10—C9—H9A	120.2
C10—N2—Cu1	132.2 (3)	N2—C10—C9	122.9 (4)
C11—N2—Cu1	110.1 (2)	N2—C10—H10A	118.5
N1—C1—C2	121.9 (4)	C9—C10—H10A	118.5
N1—C1—H1A	119.0	N2—C11—C7	123.3 (4)
C2—C1—H1A	119.0	N2—C11—C12	116.9 (3)
C3—C2—C1	120.0 (4)	C7—C11—C12	119.8 (4)
C3—C2—H2A	120.0	N1—C12—C4	122.3 (4)
C1—C2—H2A	120.0	N1—C12—C11	117.3 (3)
C2—C3—C4	119.7 (4)	C4—C12—C11	120.4 (3)
N2—Cu1—N1—C1	178.6 (4)	C6—C7—C8—C9	−179.2 (4)
$N2^{i}$—Cu1—N1—C1	−63.7 (4)	C7—C8—C9—C10	1.6 (7)
I1—Cu1—N1—C1	57.5 (4)	C11—N2—C10—C9	−1.0 (6)
N2—Cu1—N1—C12	5.0 (3)	Cu1—N2—C10—C9	178.6 (3)
$N2^{i}$—Cu1—N1—C12	122.7 (3)	C8—C9—C10—N2	−0.7 (7)
I1—Cu1—N1—C12	−116.1 (3)	C10—N2—C11—C7	2.0 (5)
$N1^{i}$—Cu1—N2—C10	−3.1 (4)	Cu1—N2—C11—C7	−177.6 (3)
N1—Cu1—N2—C10	176.6 (4)	C10—N2—C11—C12	−178.3 (3)
$N2^{i}$—Cu1—N2—C10	82.0 (3)	Cu1—N2—C11—C12	2.1 (4)
I1—Cu1—N2—C10	−98.0 (3)	C8—C7—C11—N2	−1.2 (6)

$N1^{i}$—Cu1—N2—C11	176.5 (2)	C6—C7—C11—N2	177.4 (4)
N1—Cu1—N2—C11	−3.8 (2)	C8—C7—C11—C12	179.1 (4)
$N2^{i}$—Cu1—N2—C11	−98.4 (2)	C6—C7—C11—C12	−2.3 (6)
I1—Cu1—N2—C11	81.6 (2)	C1—N1—C12—C4	−1.3 (6)
C12—N1—C1—C2	2.1 (7)	Cu1—N1—C12—C4	172.9 (3)
Cu1—N1—C1—C2	−171.2 (4)	C1—N1—C12—C11	−179.7 (4)
N1—C1—C2—C3	−0.3 (7)	Cu1—N1—C12—C11	−5.5 (4)
C1—C2—C3—C4	−2.2 (7)	C3—C4—C12—N1	−1.2 (6)
C2—C3—C4—C12	2.9 (6)	C5—C4—C12—N1	179.4 (4)
C2—C3—C4—C5	−177.8 (5)	C3—C4—C12—C11	177.2 (4)
C12—C4—C5—C6	−0.1 (7)	C5—C4—C12—C11	−2.2 (6)
C3—C4—C5—C6	−179.5 (5)	N2—C11—C12—N1	2.2 (5)
C4—C5—C6—C7	1.3 (7)	C7—C11—C12—N1	−178.1 (4)
C5—C6—C7—C11	0.0 (7)	N2—C11—C12—C4	−176.3 (3)
C5—C6—C7—C8	178.5 (5)	C7—C11—C12—C4	3.5 (5)
C11—C7—C8—C9	−0.6 (6)		

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

Fig. 1

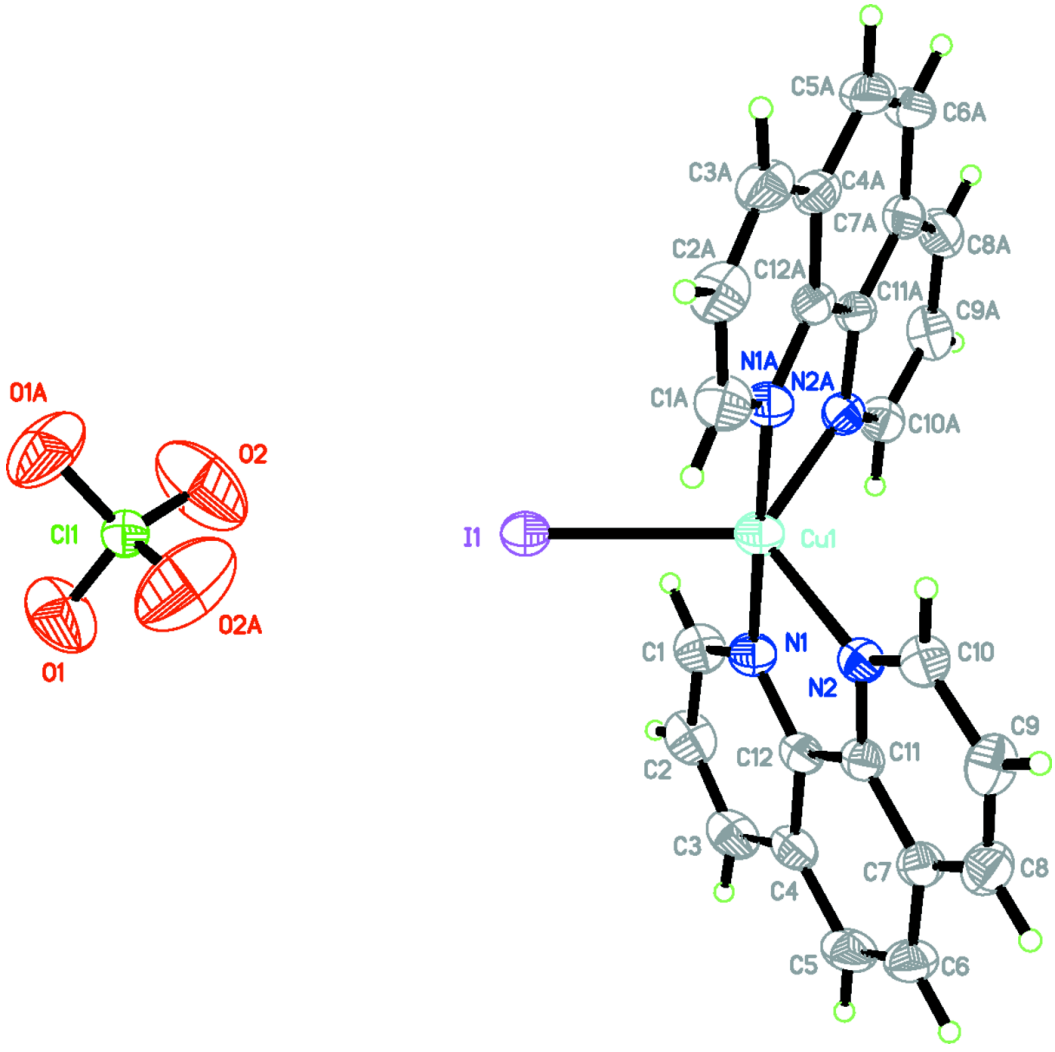
O1A
O2
Cl1
O2A
O1
C5A
C6A
C3A
C4A
C7A
C8A
C2A
C12A
C11A
C9A
N1A
N2A
C1A
C10A
I1
Cu1
C1
N1
N2
C10
C9
C2
C12
C11
C3
C7
C8
C4
C5
C6

Fig. 2