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Di-μ-iodo-bis[(4,4′-dimethyl-2,2′-bipyridine)copper(I)]

Hua Feng,* Dong-Cheng Hu, Hui-Xia Guo, Fei Zha and Chang-Qiu Hu

College of Chemistry & Chemical Engineering, Northwest Normal University, Lanzhou, Gansu 730070, People's Republic of China
Correspondence e-mail: hfeng@nwnu.edu.cn

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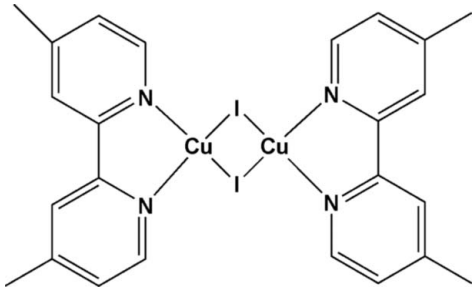
Key indicators: single-crystal X-ray study; T = 295 K; mean σ(C–C) = 0.008 Å; R factor = 0.050; wR factor = 0.129; data-to-parameter ratio = 21.2.

The title binuclear compound, $[Cu_2I_2(C_{12}H_{12}N_2)_2]$, has crystallographic mirror symmetry. The Cu atoms are monovalent and have a tetrahedral environment. The molecules pack in a layer structure.

Related literature

For related literature, see Che *et al.* (2000); Hou *et al.* (2004); Kutoglu *et al.* (1991).



Experimental

Crystal data

$[Cu_2I_2(C_{12}H_{12}N_2)_2]$
M_r = 749.35
Orthorhombic, *Pnma*
a = 11.162 (2) Å
b = 17.432 (4) Å
c = 13.794 (3) Å
V = 2684.0 (10) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 3.90 mm^{-1}
T = 295 (2) K
0.18 × 0.16 × 0.15 mm

Data collection

Bruker APEX area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Bruker, 2002)
T_{min} = 0.403, T_{max} = 0.557
15856 measured reflections
3178 independent reflections
2373 reflections with $I > 2\sigma(I)$
R_{int} = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.050
$wR(F^2)$ = 0.129
S = 1.10
3178 reflections
150 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.53 e Å^{-3}
$\Delta\rho_{min}$ = −0.78 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu1—N2	2.069 (4)	Cu1—I1	2.5948 (9)
Cu1—N1	2.074 (4)	Cu1—I2	2.6307 (9)
Cu1—Cu1^{i}	2.5274 (14)		
N2—Cu1—N1	79.37 (16)	N2—Cu1—I2	106.69 (11)
N2—Cu1—I1	116.08 (11)	N1—Cu1—I2	112.00 (12)
N1—Cu1—I1	113.27 (12)	I1—Cu1—I2	121.66 (3)

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

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LN3051).

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supplementary materials

Acta Cryst. (2007). E**63**, m2538 [**doi:10.1107/S1600536807018041**]

Di-μ-iodo-bis[(4,4'-dimethyl-2,2'-bipyridine)copper(I)]

H. Feng, D.-C. Hu, H.-X. Guo, F. Zha and C.-Q. Hu

Comment

4,4'-Dimethyl-2,2'-bipyridine, a commercially available bidentate chelating heterocyclic ligand, furnishes complexes from a large range of metal salts. The structure of the binuclear title compound, (I), obtained from copper(I) iodide is shown in Fig. 1. Each Cu atom is chelated by the heterocycle and two μ_2-I atoms in a four-coordinate environment and shows tetrahedron geometry (Fig. 1). The two Cu—I bond lengths [2.5948 (9) and 2.6307 (9) Å] are comparable with those reported earlier (Kutoglu *et al.*, 1991). The molecule has crystallographic mirror

symmetry and the structure is a binuclear layer compound. The two copper(I) atoms are separated by a distance of 2.5274 (14) Å indicating a strong $Cu^{I}\cdots Cu^{I}$ interaction, which is comparable with the $Cu^{I}\cdots Cu^{I}$ distance found previously (Che *et al.*, 2000; Hou *et al.*, 2004).

Experimental

4,4'-dimethyl-2,2'-bipyridine was commercially available and was used as received without further purification. This compound (0.0184 g, 0.1 mmol), together with a saturated potassium iodide solution containing copper(I) iodide (0.0190 g, 0.1 mmol), were dissolved in water (10 ml). Then the solution was placed and sealed in a 15 ml Teflon-lined stainless steel reactor and heated to 453 K for 72 h, then cooled down to room temperature at a rate of 5 K/h. Red block crystals were formed in about 50% yield.

Refinement

H atoms were placed in calculated positions (C—H 0.93 Å; U_{iso}(H) = 1.2U_{eq}(C) for the ring H atoms and C—H 0.96 Å; U_{iso}(H) = 1.5U_{eq}(C) for the methyl groups) and were included in the refinement in the riding model approximation. The largest peak in the final difference map was 0.95 Å from atom I2.

Figures

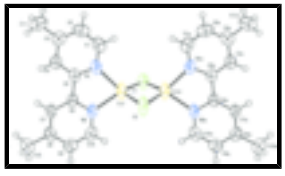


Fig. 1. A view of the molecule of (I) showing the atom labeling scheme. Displacement ellipsoids are drawn at the 50% probability level, and H atoms are drawn as spheres of arbitrary radii. Atoms labeled with "a" are generated by the symmetry operation x, 1/2 − y, z.

Di-μ-iodo-bis[(4,4'-dimethyl-2,2'-bipyridine)copper(I)]

Crystal data

$[Cu_2I_2(C_{12}H_{12}N_2)_2]$	F_{000} = 1440
M_r = 749.35	D_x = 1.854 Mg m^{-3}
Orthorhombic, *Pnma*	Mo *K*α radiation λ = 0.71073 Å
Hall symbol: -P 2ac 2n	Cell parameters from 2945 reflections
a = 11.162 (2) Å	θ = 2.2–24.9º
b = 17.432 (4) Å	μ = 3.90 mm^{-1}
c = 13.794 (3) Å	*T* = 295 (2) K
V = 2684.0 (10) Å^3	Block, red
Z = 4	0.18 × 0.16 × 0.15 mm

Data collection

Bruker APEX area-detector diffractometer	3178 independent reflections
Radiation source: fine-focus sealed tube	2373 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.031
T = 295(2) K	θ_{max} = 27.5º
φ and ω scans	θ_{min} = 1.9º
Absorption correction: multi-scan (SADABS; Bruker, 2002)	*h* = −14→14
T_{min} = 0.403, T_{max} = 0.557	*k* = −21→22
15856 measured reflections	*l* = −8→17

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)]$ = 0.050	H-atom parameters constrained
$wR(F^2)$ = 0.129	$w = 1/[\sigma^2(F_o^2) + (0.0571P)^2 + 3.2733P]$ where $P = (F_o^2 + 2F_c^2)/3$
S = 1.10	$(\Delta/\sigma)_{max}$ = 0.001
3178 reflections	$\Delta\rho_{max}$ = 1.53 e Å^{-3}
150 parameters	$\Delta\rho_{min}$ = −0.78 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	0.00472 (7)	0.32249 (4)	0.35891 (5)	0.0652 (2)
I1	0.02234 (5)	0.2500	0.19524 (4)	0.05818 (19)
I2	−0.04114 (7)	0.2500	0.52201 (4)	0.0760 (2)
N1	0.1340 (4)	0.4068 (2)	0.3767 (3)	0.0523 (10)
N2	−0.1010 (4)	0.4200 (2)	0.3583 (3)	0.0484 (10)
C1	0.2528 (5)	0.3975 (4)	0.3846 (4)	0.0640 (15)
H1A	0.2840	0.3481	0.3809	0.077*
C2	0.3299 (5)	0.4571 (4)	0.3978 (4)	0.0695 (16)
H2A	0.4116	0.4474	0.4025	0.083*
C3	0.2893 (5)	0.5313 (3)	0.4042 (4)	0.0603 (14)
C4	0.1643 (5)	0.5412 (3)	0.3960 (3)	0.0519 (12)
H4A	0.1312	0.5900	0.4006	0.062*
C5	0.0911 (4)	0.4788 (3)	0.3812 (3)	0.0436 (10)
C6	−0.0402 (4)	0.4862 (3)	0.3683 (3)	0.0428 (10)
C7	−0.0989 (5)	0.5568 (3)	0.3662 (3)	0.0493 (12)
H7A	−0.0552	0.6019	0.3730	0.059*
C8	−0.2214 (5)	0.5600 (3)	0.3542 (4)	0.0554 (13)
C9	−0.2824 (5)	0.4923 (3)	0.3449 (4)	0.0591 (14)
H9A	−0.3651	0.4923	0.3370	0.071*
C10	−0.2196 (5)	0.4238 (3)	0.3473 (4)	0.0554 (13)
H10A	−0.2622	0.3782	0.3410	0.066*
C11	0.3689 (6)	0.5995 (4)	0.4184 (5)	0.090 (2)
H11A	0.4424	0.5836	0.4484	0.136*
H11B	0.3293	0.6362	0.4593	0.136*
H11C	0.3859	0.6225	0.3567	0.136*
C12	−0.2839 (6)	0.6367 (3)	0.3491 (5)	0.0819 (19)
H12A	−0.2307	0.6741	0.3216	0.123*
H12B	−0.3067	0.6525	0.4132	0.123*
H12C	−0.3541	0.6323	0.3093	0.123*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Cu1	0.0990 (6)	0.0276 (3)	0.0688 (5)	0.0008 (3)	0.0047 (4)	−0.0003 (3)
I1	0.0780 (4)	0.0420 (3)	0.0545 (3)	0.000	0.0019 (2)	0.000
I2	0.1387 (6)	0.0316 (3)	0.0577 (4)	0.000	0.0104 (3)	0.000
N1	0.069 (3)	0.038 (2)	0.051 (2)	0.0070 (19)	0.003 (2)	0.0018 (18)

N2	0.067 (3)	0.0310 (19)	0.048 (2)	−0.0040 (18)	0.0057 (19)	−0.0018 (16)
C1	0.076 (4)	0.060 (3)	0.056 (3)	0.017 (3)	−0.002 (3)	0.002 (3)
C2	0.051 (3)	0.099 (5)	0.058 (4)	0.011 (3)	−0.004 (3)	0.005 (3)
C3	0.064 (4)	0.066 (4)	0.051 (3)	−0.006 (3)	0.000 (2)	0.000 (3)
C4	0.066 (3)	0.042 (3)	0.048 (3)	−0.003 (2)	0.004 (2)	−0.003 (2)
C5	0.060 (3)	0.037 (2)	0.034 (2)	0.003 (2)	0.002 (2)	0.0001 (18)
C6	0.060 (3)	0.034 (2)	0.035 (2)	−0.001 (2)	0.004 (2)	0.0002 (18)
C7	0.062 (3)	0.032 (2)	0.054 (3)	−0.001 (2)	0.003 (2)	−0.005 (2)
C8	0.064 (3)	0.049 (3)	0.052 (3)	0.008 (2)	0.005 (2)	0.000 (2)
C9	0.052 (3)	0.068 (4)	0.057 (3)	−0.005 (3)	0.007 (2)	0.003 (3)
C10	0.068 (4)	0.048 (3)	0.050 (3)	−0.013 (3)	0.003 (3)	0.000 (2)
C11	0.064 (4)	0.106 (5)	0.101 (5)	−0.024 (4)	−0.003 (4)	−0.006 (4)
C12	0.079 (4)	0.062 (4)	0.105 (5)	0.020 (3)	0.002 (4)	−0.002 (4)

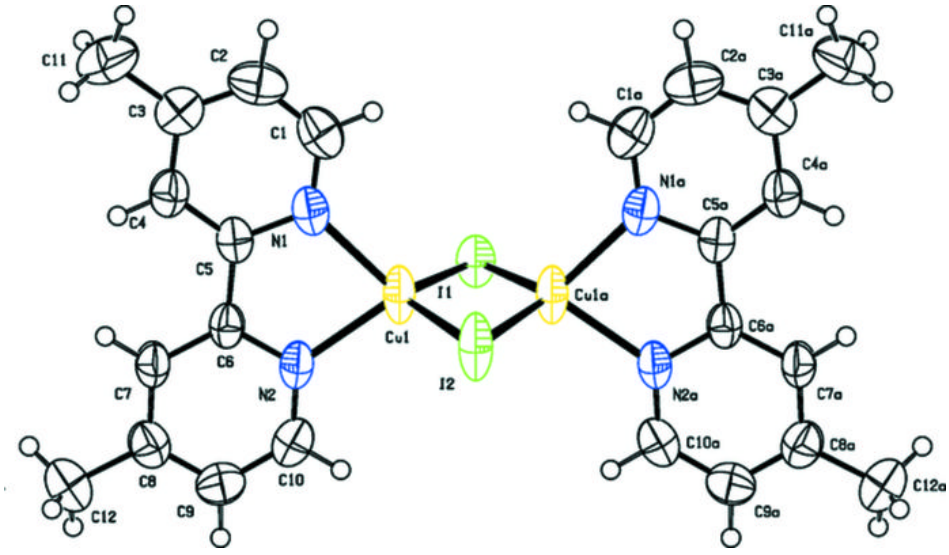
Geometric parameters (Å, °)

Cu1—N2	2.069 (4)	C4—H4A	0.9300
Cu1—N1	2.074 (4)	C5—C6	1.482 (7)
Cu1—Cu1^{i}	2.5274 (14)	C6—C7	1.394 (6)
Cu1—I1	2.5948 (9)	C7—C8	1.379 (7)
Cu1—I2	2.6307 (9)	C7—H7A	0.9300
N1—C1	1.341 (7)	C8—C9	1.369 (7)
N1—C5	1.345 (6)	C8—C12	1.509 (7)
N2—C10	1.334 (7)	C9—C10	1.385 (7)
N2—C6	1.347 (6)	C9—H9A	0.9300
C1—C2	1.361 (8)	C10—H10A	0.9300
C1—H1A	0.9300	C11—H11A	0.9600
C2—C3	1.374 (8)	C11—H11B	0.9600
C2—H2A	0.9300	C11—H11C	0.9600
C3—C4	1.411 (8)	C12—H12A	0.9600
C3—C11	1.497 (8)	C12—H12B	0.9600
C4—C5	1.375 (6)	C12—H12C	0.9600
N2—Cu1—N1	79.37 (16)	N1—C5—C4	122.2 (5)
N2—Cu1—Cu1^{i}	145.23 (12)	N1—C5—C6	115.4 (4)
N1—Cu1—Cu1^{i}	135.10 (11)	C4—C5—C6	122.4 (4)
N2—Cu1—I1	116.08 (11)	N2—C6—C7	121.2 (4)
N1—Cu1—I1	113.27 (12)	N2—C6—C5	115.8 (4)
Cu1^{i}—Cu1—I1	60.855 (17)	C7—C6—C5	123.0 (4)
N2—Cu1—I2	106.69 (11)	C8—C7—C6	120.3 (5)
N1—Cu1—I2	112.00 (12)	C8—C7—H7A	119.8
Cu1^{i}—Cu1—I2	61.290 (17)	C6—C7—H7A	119.8
I1—Cu1—I2	121.66 (3)	C9—C8—C7	118.0 (5)
Cu1—I1—Cu1^{i}	58.29 (3)	C9—C8—C12	122.0 (5)
Cu1^{i}—I2—Cu1	57.42 (3)	C7—C8—C12	120.0 (5)
C1—N1—C5	117.5 (5)	C8—C9—C10	119.4 (5)
C1—N1—Cu1	127.8 (4)	C8—C9—H9A	120.3
C5—N1—Cu1	114.8 (3)	C10—C9—H9A	120.3

C10—N2—C6	118.0 (4)	N2—C10—C9	123.2 (5)
C10—N2—Cu1	127.4 (3)	N2—C10—H10A	118.4
C6—N2—Cu1	114.6 (3)	C9—C10—H10A	118.4
N1—C1—C2	122.9 (5)	C3—C11—H11A	109.5
N1—C1—H1A	118.5	C3—C11—H11B	109.5
C2—C1—H1A	118.5	H11A—C11—H11B	109.5
C1—C2—C3	121.3 (5)	C3—C11—H11C	109.5
C1—C2—H2A	119.3	H11A—C11—H11C	109.5
C3—C2—H2A	119.3	H11B—C11—H11C	109.5
C2—C3—C4	115.8 (5)	C8—C12—H12A	109.5
C2—C3—C11	124.1 (6)	C8—C12—H12B	109.5
C4—C3—C11	120.0 (5)	H12A—C12—H12B	109.5
C5—C4—C3	120.2 (5)	C8—C12—H12C	109.5
C5—C4—H4A	119.9	H12A—C12—H12C	109.5
C3—C4—H4A	119.9	H12B—C12—H12C	109.5
N2—Cu1—I1—Cu1^{i}	−140.73 (13)	C1—C2—C3—C11	−179.8 (6)
N1—Cu1—I1—Cu1^{i}	130.02 (12)	C2—C3—C4—C5	−0.8 (8)
I2—Cu1—I1—Cu1^{i}	−8.08 (5)	C11—C3—C4—C5	178.8 (5)
N2—Cu1—I2—Cu1^{i}	144.44 (12)	C1—N1—C5—C4	−1.7 (7)
N1—Cu1—I2—Cu1^{i}	−130.53 (12)	Cu1—N1—C5—C4	177.8 (4)
I1—Cu1—I2—Cu1^{i}	8.04 (4)	C1—N1—C5—C6	177.7 (4)
N2—Cu1—N1—C1	−179.2 (5)	Cu1—N1—C5—C6	−2.7 (5)
Cu1^{i}—Cu1—N1—C1	6.2 (5)	C3—C4—C5—N1	1.8 (7)
I1—Cu1—N1—C1	−65.2 (5)	C3—C4—C5—C6	−177.6 (4)
I2—Cu1—N1—C1	77.0 (5)	C10—N2—C6—C7	−0.7 (7)
N2—Cu1—N1—C5	1.4 (3)	Cu1—N2—C6—C7	178.0 (3)
Cu1^{i}—Cu1—N1—C5	−173.3 (2)	C10—N2—C6—C5	179.4 (4)
I1—Cu1—N1—C5	115.3 (3)	Cu1—N2—C6—C5	−1.9 (5)
I2—Cu1—N1—C5	−102.5 (3)	N1—C5—C6—N2	3.1 (6)
N1—Cu1—N2—C10	178.9 (4)	C4—C5—C6—N2	−177.5 (4)
Cu1^{i}—Cu1—N2—C10	−7.7 (5)	N1—C5—C6—C7	−176.8 (4)
I1—Cu1—N2—C10	68.1 (4)	C4—C5—C6—C7	2.7 (7)
I2—Cu1—N2—C10	−71.1 (4)	N2—C6—C7—C8	0.3 (7)
N1—Cu1—N2—C6	0.3 (3)	C5—C6—C7—C8	−179.8 (4)
Cu1^{i}—Cu1—N2—C6	173.7 (2)	C6—C7—C8—C9	0.2 (8)
I1—Cu1—N2—C6	−110.5 (3)	C6—C7—C8—C12	−178.2 (5)
I2—Cu1—N2—C6	110.3 (3)	C7—C8—C9—C10	−0.3 (8)
C5—N1—C1—C2	0.6 (8)	C12—C8—C9—C10	178.1 (5)
Cu1—N1—C1—C2	−178.8 (4)	C6—N2—C10—C9	0.6 (7)
N1—C1—C2—C3	0.3 (9)	Cu1—N2—C10—C9	−177.9 (4)
C1—C2—C3—C4	−0.2 (8)	C8—C9—C10—N2	−0.1 (8)

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

Fig. 1


C11
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C1
C4
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C6
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C8
C10
C12
C9
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I2
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C1a
C2a
C3a
C11a
C4a
C5a
C6a
C7a
N2a
C10a
C8a
C12a
C9a