943 reflections
69 parameters
H atoms: see below
$w = 1/[\sigma^2(F_o^2) + (0.0125P)^2 + 4.788P]$
where $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)*

$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$

	x	y	z	U_{eq}
Ni	1/3	2/3	−1/3	0.0294 (2)
I1	0	0	−0.42824 (2)	0.05254 (13)
I2	0	0	−0.25984 (2)	0.05009 (12)
S	0.29182 (7)	0.41669 (7)	−0.25930 (4)	0.0378 (2)
O	0.3912 (2)	0.5581 (2)	−0.27545 (9)	0.0374 (4)
C1	0.3264 (4)	0.3198 (3)	−0.3153 (2)	0.0496 (8)
C2	0.3498 (4)	0.3834 (4)	−0.1833 (2)	0.0549 (9)

Table 2. *Selected geometric parameters (Å, °)*

Ni—O	2.077 (2)	S—O	1.521 (2)
I1—I1^{i}	2.848 (1)	S—C1	1.784 (4)
I1—I2	3.342 (1)	S—C2	1.780 (4)
O—Ni—O^{ii}	92.37 (7)	O—S—C2	104.5 (2)
O^{iv}—Ni—O	180	O—S—C1	106.0 (2)
O—Ni—O^{v}	87.63 (7)	C1—S—C2	98.5 (2)
I1^{i}—I1—I2	180	Ni—O—S	118.9 (1)

Symmetry codes: (i) $-x, -y, -1-z$; (ii) $-x+y, 1-x, z$; (iv) $\frac{2}{3}-x, \frac{4}{3}-y, -\frac{2}{3}-z$; (v) $y-\frac{1}{3}, \frac{1}{3}-x+y, -\frac{2}{3}-z$.

The structure was solved by direct methods and expanded using Fourier techniques. All H atoms were found in difference Fourier maps and refined isotropically.

Data collection: *SMART* (Siemens, 1996). Cell refinement: *SMART* and *SAINT* (Siemens, 1996). Data reduction: *SAINT*. Program(s) used to solve structure: *SHELXTL* (Siemens, 1994). Program(s) used to refine structure: *SHELXTL*. Molecular graphics: *SHELXTL*. Software used to prepare material for publication: *SHELXTL*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1277). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1999). C55, 341–343

Di-μ-iodo-1:2κ^4*I*-bis(quinoline)-1κ*N*,2κ*N*-bis(triphenylphosphine)-1κ*P*,2κ*P*-dicopper(I)

QIONG-HUA JIN,[a] YU-XIAN WANG[a] AND XIU-LAN XIN[b]

[a]*Department of Chemistry, Capital Normal University, Beijing 100037, People's Republic of China, and* [b]*Department of Chemical Engineering, Beijing Institute of Light Industry, Beijing 100037, People's Republic of China. E-mail: jinqh@mailhost.cnu.edu.cn*

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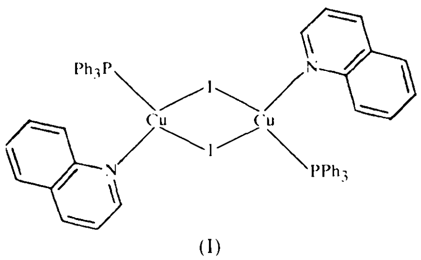
Abstract

The title compound, $[CuI(PPh_3)(C_9H_7N)]_2$ or $[Cu_2I_2(C_9H_7N)_2(C_{18}H_{15}P)_2]$, is an inversion-symmetric dimer with two Cu atoms each tetrahedrally coordinated to two I, one N and one P atom. The diamond-shaped central Cu_2I_2 group has longer I···I [4.351 (1) Å] and shorter Cu···Cu distances [3.144 (1) Å] than the $X\cdots X$ (X = Br, I) and Cu···Cu distances found in the related compounds $[CuBr(PPh_3)(C_9H_7N)]_2$ and $[CuI(C_9H_7N)_2]_2$.

Comment

Recently, we obtained the group I*B* metal complexes $[CuBr(PPh_3)(C_9H_7N)]_2$ [(II); Jin, Long *et al.*, 1998] and $[CuI(PPh_3)(phen)]$ (Jin, Xin *et al.*, 1998), where phen is 1,10-phenanthroline, which have found use in our work on the synthesis of Mo(W)–Cu(Ag)–S compounds (Hou *et al.*, 1996). We report here the structure of another such Cu^{I} complex, namely, $[CuI(PPh_3)(C_9H_7N)]_2$, (I).



(I)

The title compound consists of inversion-symmetric dimers with a diamond-shaped Cu_2I_2 group at the center. The Cu atom is coordinated to two I atoms, one N atom and one P atom in a distorted tetrahedral arrangement.

The I—Cu—I angle [108.30 (2)°] in (I) is larger than the Br—Cu—Br angle [95.83 (2)°] in (II) and the I—Cu—I angle [102.4 (1)°] in $[CuI(C_9H_7N)_2]_2$, (III) (Rath *et al.*, 1986), while the Cu—I—Cu angle [71.70 (2)°] in (I) is smaller than the Cu—Br—Cu angle [84.17 (2)°] in (II) and the Cu—I—Cu angle [77.6 (1)°] in (III). These trends in bond angles lead to a shorter Cu···Cu separation [3.144 (1) Å] in (I) than in both (II) [3.414 (1) Å] and (III) [3.364 (5) Å], and to a longer I···I distance [4.351 (1) Å] than the Br···Br distance [3.780 (5) Å] in (II) and the I···I distance [4.188 (2) Å] in (III).

The average Cu—I distance [2.683 (9) Å] agrees well with the Cu—I distances [2.686 (4) Å] found in similar dimeric molecules, *e.g.* (III).

The Cu—P [2.246 (6) Å] and Cu—N distances [2.097 (3) Å] are also in good agreement with values found in similar compounds containing P and N atoms, *e.g.* (II) [Cu—P 2.2160 (12) and Cu—N 2.065 (3) Å], $[CuI(PPh_3)(phen)]$ [Cu—P 2.1977 (9), and Cu—N 2.071 (3) and 2.111 (3) Å; Jin, Xin *et al.*, 1998], $[Cu_2Cl_2(4,4'\text{-bipy})(PPh_3)_2]$ [Cu—P 2.199 (1) and Cu—N 2.057 (3) Å; Lu *et al.*, 1997], where 4,4′-bipy is 4,4′-bipyridine.

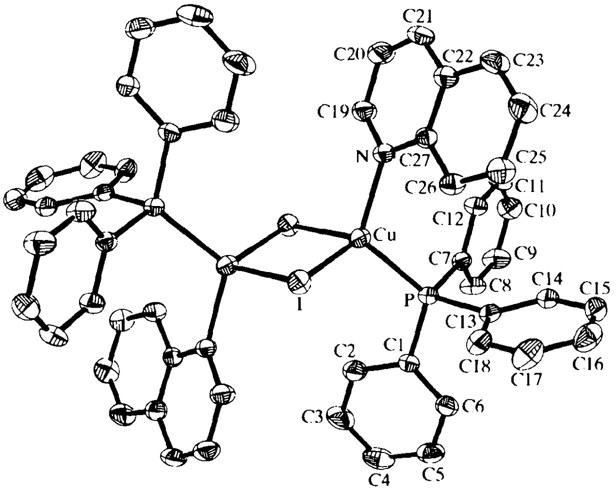


Fig. 1. View of the title complex, with displacement ellipsoids shown at the 30% probability level.

Experimental

$[CuI(PPh_3)(C_9H_7N)]_2$ was obtained by the reaction of CuI and PPh_3 (molar ratio 1:2) in the presence of quinoline in DMF solution at 343 K. Yellow prismatic crystals were produced by slow evaporation of the solution.

Crystal data

$[Cu_2I_2(C_9H_7N)_2(C_{18}H_{15}P)_2]$
$M_r = 1163.74$
Triclinic
$P\bar{1}$
$a = 9.304\,(2)$ Å
$b = 10.9792\,(13)$ Å
$c = 13.685\,(3)$ Å
$\alpha = 107.896\,(10)°$
$\beta = 107.812\,(14)°$
$\gamma = 96.383\,(11)°$
$V = 1233.7\,(4)$ Å^3
$Z = 1$
$D_x = 1.566$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 30 reflections
$\theta = 5.62$–$14.75°$
$\mu = 2.215$ mm^{-1}
$T = 293\,(2)$ K
Prism
0.47 × 0.30 × 0.18 mm
Yellow

Data collection

Siemens *P*4 diffractometer
θ–2θ scans
Absorption correction: ψ scan (*XEMP*; Siemens, 1991)
$T_{min} = 0.487$, $T_{max} = 0.671$
5134 measured reflections
4263 independent reflections
3444 reflections with $I > 2\sigma(I)$

$R_{int} = 0.018$
$\theta_{max} = 25°$
$h = -1 \rightarrow 10$
$k = -12 \rightarrow 12$
$l = -16 \rightarrow 15$
3 standard reflections every 97 reflections intensity decay: 2.43%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.029$
$wR(F^2) = 0.065$
$S = 1.023$
4263 reflections
282 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0392P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{max} = -0.002$
$\Delta\rho_{max} = 0.591$ e Å^{-3}
$\Delta\rho_{min} = -0.487$ e Å^{-3}
Extinction correction: *SHELXL*93
Extinction coefficient: 0.0140 (6)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °)*

I—Cu	2.6617 (6)	Cu—N	2.097 (3)
I—Cu^i	2.7059 (5)	Cu—P	2.2466 (11)
Cu—I—Cu^i	71.70 (2)	N—Cu—I^i	103.46 (7)
N—Cu—P	117.20 (8)	P—Cu—I^i	106.07 (3)
N—Cu—I	106.25 (8)	I—Cu—I^i	108.30 (2)
P—Cu—I	114.70 (3)		

Symmetry code: (i) $1 - x, 2 - y, 2 - z$.

All H atoms were placed geometrically and refined in riding mode with isotropic displacement parameters 20% greater than those of the parent atoms.

Data collection: *XSCANS* (Fait, 1991). Cell refinement: *XSCANS*. Data reduction: *XSCANS*. Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL*93 (Sheldrick, 1993). Molecular graphics: *SHELXTL* (Siemens, 1994). Software used to prepare material for publication: *SHELXL*93.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: JZ1317). Services for accessing these data are described at the back of the journal.

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Poly[mercury(II)-μ-4,4′-bipyridine-di-μ-bromo]

LONG PAN,[a] NENGWU ZHENG,[a] YONGGANG WU,[a] SHICHUN WU[b] AND XIAOYING HUANG[c]

[a]*Department of Chemistry, University of Science & Technology of China, Hefei Anhui 230026, People's Republic of China,* [b]*New Drug Research of Anhui Provincial Institute For Drug Control, Hefei Anhui 230061, People's Republic of China, and* [c]*Fujian Institute of Research on the Structure of Matter, Chinese Academy of Science, Fuzhou, Fujian 350002, People's Republic of China. E-mail: lpan@mail.ach.ustc.edu.cn*

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Abstract

The title compound, $[HgBr_2(C_{10}H_8N_2)]_n$, was obtained by mixing equimolar ethanolic solutions of mercuric bromide and 4,4′-bipyridine (4,4′-bipy). The bipy ligand acts as a linear bifunctional bridge forming a planar $\{-[Hg(4,4'\text{-bipy})]-\}_n$ belt in the direction of the *a* axis. The remaining mercury coordination sites are occupied by Br^- ions which link Hg centres in neighbouring belts *via* double bridges to form extended two-dimensional layers.

Comment

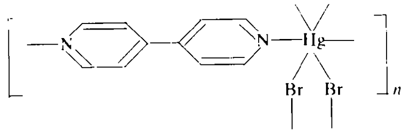
There has been significant interest recently in metal complexes containing 4,4′-bipyridine (4,4′-bipy). The rod-like rigidity of this ligand leads to metal complexes which are often extended solids with topological structures. Three types of polymer structure formed by 4,4′-bipy can be summarized as follows:

(i) each metal ion is connected by two bridging 4,4′-bipy ligands to form one-dimensional $[M(4,4'\text{-bipy})]_n$ polymeric chains, for example, $\{[Ni(4,4'\text{-bipy})(C_5H_9OS_2)_2]\cdot 2CCl_4\}_n$ (Gable *et al.*, 1985), $[Cd(4,4'\text{-bipy})(C_4H_{17}OS_2)_2]_n$ (Abrahams *et al.*, 1990), $[Cu(4,4'\text{-bipy})(H_2O)_2(ClO_4)_2]_n\cdot n(4,4'\text{-bipy})$ (Chen *et al.*, 1996), $[Cu(4,4'\text{-bipy})(2,2'\text{-bipy})(ClO_4)_2]_n$ (Chen *et al.*, 1992) and $[Co(NCS)_2(H_2O)_2(4,4'\text{-bipy})]\cdot(4,4'\text{-bipy})$ (Lu *et al.*, 1997). In the last two of these, the one-dimensional chains are connected by hydrogen bonding involving uncoordinated 4,4′-bipy, resulting in a two-dimensional structure.

(ii) Mutually interpenetrating two-dimensional sheets of $[Cu(4,4'\text{-bipy})Cl]$ were synthesized by Yaghi & Li (1995) and two-dimensional layers were found in $\{[Cd(4,4'\text{-bipy})_2](NO_3)_2\}_n$ (Fujita *et al.*, 1994) and $[Co(NCS)_2(4,4'\text{-bipy})_2]\cdot 2[(CH_3CH_2)_2O]$ (Lu *et al.*, 1997), in which the metal ion was coordinated by four bridging 4,4′-bipy ligands. Infinite square-grid two-dimensional cationic sheets of composition $[Cd(H_2O)_2(4,4'\text{-bipy})_2]_n^{2n+}$ were observed in $[Cd(H_2O)_2(4,4'\text{-bipy})_2]PF_6\cdot 2(4,4'\text{-bipy})\cdot 4H_2O$ (Robson *et al.*, 1992).

(iii) Three-dimensional structures with super-diamond-type nets are observed in $[Cu(4,4'\text{-bipy})_{1.5}]NO_3\cdot 1.25H_2O$ (Yaghi & Li, 1995) and $[Cu(4,4'\text{-bipy})_2(PF_6)]$ (MacGillivray *et al.*, 1994), the extent of interpenetration leaving extended channels through the structure. However, in $[Zn(4,4'\text{-bipy})_2(SiF_6)]\cdot 2H_2O$ (Gable *et al.*, 1990), there are no accessible voids. Moreover, the complex $[Ag(4,4'\text{-bipy})_2(CF_3SO_3)]_n$ (Carlucci *et al.*, 1994) is also of this type. Three-dimensional structures with large square channels are found in $[Zn(4,4'\text{-bipy})_2(SiF_6)]_n\cdot DMF$ (Subramanian & Zaworotko, 1995) and $[Zn_2(4,4'\text{-bipy})(PO_3F)_2]$ (Halasyamani *et al.*, 1997). The three-dimensional framework of $[Ag(4,4'\text{-bipy})]NO_3$ (Robinson & Zaworotko, 1995) is assembled from T-shaped cationic building blocks.

The title compound, (I), belongs to type (ii). The Hg^{II} ion is coordinated by two N atoms from two different 4,4′-bipy ligands and by four Br^- ligands (Fig. 1). The N_2Br_4 coordination about mercury is close to octahedral (Table 2).



(I)