metal-organic papers

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Key indicators

Single-crystal X-ray study T = 298 KMean σ (C–C) = 0.016 Å Disorder in solvent or counterion R factor = 0.054 wR factor = 0.166 Data-to-parameter ratio = 14.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

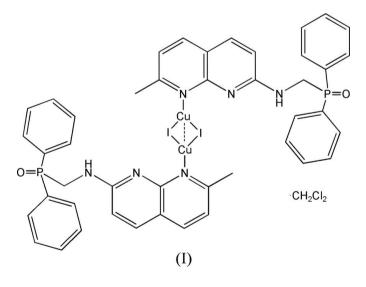
Di- μ -iodo-bis({2-[(diphenylphosphoryl)methylamino]-7-methyl-1,8-naphthyridine- κN^8 }copper(I)) dichloromethane solvate

The title complex, $[Cu_2I_2(C_{22}H_{20}N_3OP)_2]\cdot CH_2Cl_2$, has a dimeric structure with a central Cu_2I_2 core formed about a twofold rotation axis. Each Cu^I centre is coordinated by an N atom at the 8-position of 1,8-naphthyridine to give an approximately planar triangular coordination geometry. Metal-metal interactions are also present, with a $Cu \cdot \cdot Cu$ distance of 2.539 (2) Å. Complex molecules are connected into chains *via* intermolecular $N-H \cdot \cdot \cdot O$ hydrogen bonds.

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Comment

1,8-Naphthyridine (napy) ligands are versatile rigid ligands that have been used for the construction of mononuclear (Pastene *et al.*, 2000), binuclear (Ziessel *et al.*, 2000; He & Lippard, 2000) and multinuclear complexes (Chan *et al.*, 1998). Some Cu^I complexes with napy ligands have already been reported (Maekawa *et al.*, 1998; Scott *et al.*, 1998), although there have been none to date containing halide anions.



The title complex, (I) (Fig. 1), comprises twofold-symmetric dinuclear Cu^{I} complexes, with two 2-*N*-(diphenylphosphoryl)methylamino-7-methyl-1,8-naphthyridine (dnpa) ligands bound to two Cu^{I} atoms, which are bridged by I atoms. Of the three potential coordinating N atoms in the dpna ligand, only N1 is coordinated to Cu1. Several different coordination modes have previously been described for related Cu^{I} -napy complexes (Chan *et al.*, 1998). The Cu \cdots Cu distance of 2.539 (2) Å is relatively short for this type of compound (Yim *et al.*, 2003).

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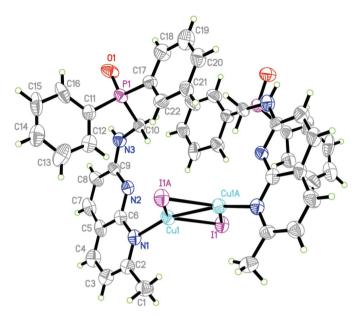


Figure 1

The molecular structure of (I), showing displacement ellipsoids at the 30% probability level. The solvent molecule is not shown. [Symmetry code: (A) $1 - x, y, -z + \frac{1}{2}$.]

Complex molecules of (I) are connected via intermolecular N-H···O hydrogen bonds into chains running along the cdirection (Fig. 2 and Table 2).

Experimental

2-N-(Diphenylphosphoryl)methylamino-7-methyl-1,8-naphthyridine (dpna) was prepared according to the literature method of Durran et al. (2000). An equimolar mixture of dpna (37 mg, 0.1 mmol) and CuI (19 mg, 0.1 mmol) in dichloromethane (30 ml) was stirred for 6 h at room temperature. The resulting solution was filtered and concentrated to 5 ml. Addition of diethyl ether (20 ml) gave the product, (I), as a yellow solid. Recrystallization by slow diffusion of diethyl ether into a solution of the crude product in dichloromethane gave yellow crystals of (I).

Crystal data

$[Cu_2I_2(C_{22}H_{20}N_3OP)_2]\cdot CH_2Cl_2$
$M_r = 1212.59$
Monoclinic, $C2/c$
a = 19.613 (4) Å
b = 16.257 (3) Å
c = 18.031 (4) Å
$\beta = 122.203 \ (3)^{\circ}$
V = 4864.7 (16) Å ³

Data collection

Bruker SMART APEX CCD areadetector diffractometer ω scans Absorption correction: multi-scan (SADABS; Bruker, 2000) $T_{\rm min} = 0.645, \ T_{\rm max} = 0.734$

Z = 4 $D_x = 1.656 \text{ Mg m}^{-3}$ Mo $K\alpha$ radiation $\mu = 2.37 \text{ mm}^-$ T = 298 (2) K Block, yellow $0.29 \times 0.16 \times 0.13~\text{mm}$

12486 measured reflections 4229 independent reflections 3001 reflections with $I > 2\sigma(I)$ $R_{\rm int}=0.054$ $\theta_{\rm max} = 25.0^\circ$

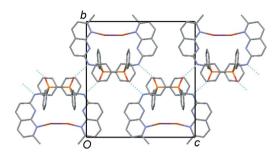


Figure 2

A projection of (I) along the *a* axis, showing hydrogen-bonded chains running along c. N-H···O hydrogen bonds are shown as dashed lines. H atoms have been omitted.

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0828P)^2]$
$R[F^2 > 2\sigma(F^2)] = 0.054$	+ 10.2228P]
$wR(F^2) = 0.166$	where $P = (F_0^2 + 2F_c^2)/3$
S = 1.00	$(\Delta/\sigma)_{\rm max} = 0.001$
4229 reflections	$\Delta \rho_{\rm max} = 0.67 \ {\rm e} \ {\rm \AA}^{-3}$
285 parameters	$\Delta \rho_{\rm min} = -0.72 \text{ e} \text{ \AA}^{-3}$
H-atom parameters constrained	

Table 1

Selected	geometric	parameters	(Å,	°).
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Cu1-N1	1.991 (7)	Cu1-I1 ⁱ	2.5481 (13)	
$Cu1 \cdots Cu1^i$	2.539 (2)	Cu1-I1	2.6187 (14)	
N1-Cu1-I1 ⁱ	124.5 (2)	I1 ⁱ -Cu1-I1	121.15 (4)	
N1-Cu1-I1	112.6 (2)			

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

Table 2

Hydrogen-bond geometry (Å, °).

$D - H \cdot \cdot \cdot A$	D-H	$H \cdot \cdot \cdot A$	$D \cdots A$	$D - \mathbf{H} \cdots A$
$N3-H3\cdots O1^{ii}$	0.86	2.07	2.887 (3)	160
Symmetry code: (ii)	$-r \pm 1 - v - 7$	上1		

Symmetry code: (ii) -x + 1, -y, -z + 1.

All H atoms were placed in calculated positions and allowed to ride during subsequent refinement, with C-H distances of 0.93-0.96 Å and an N-H distance of 0.86 Å, and with $U_{iso}(H) =$ $1.2U_{eq}(C,N)$. The dichloromethane molecule is disordered around a twofold rotation axis with equal occupancy factors of 0.5 for the two components, and is modelled with one of its Cl atoms lying on the twofold axis. The C-Cl and Cl···Cl distances were restrained to ensure a reasonable geometry. The refined displacement parameters are large, which may indicate partial solvent loss. The highest peak and deepest hole in the difference density map are associated with the disordered CH₂Cl₂ molecule.

Data collection: SMART (Bruker, 2000); cell refinement: SAINT (Bruker, 2000); data reduction: SAINT; program(s) used to solve structure: SHELXS97 (Sheldrick, 1997); program(s) used to refine structure: SHELXL97 (Sheldrick, 1997); molecular graphics: SHELXTL (Bruker, 2000); software used to prepare material for publication: SHELXTL.

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