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Dichlorido(hexamethylphosphamide)(morpholine-κ*N*)copper(II) at 153 K

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In the title compound, [$CuCl_2(C_4H_9NO)(C_6H_{18}N_3OP)$], the Cu atom is situated in a slightly distorted tetrahedral coordination geometry. The H—N bond of morpholine is in the axial position.

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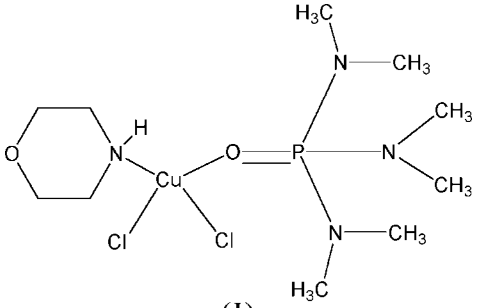
Key indicators

Single-crystal X-ray study
T = 153 K
Mean σ(C–C) = 0.002 Å
R factor = 0.024
wR factor = 0.063
Data-to-parameter ratio = 21.0

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

Comment

There are numerous hexamethylphosphamide (HMPA) complexes, such as $Nb^{III}Cl_3(OR)_2$(HMPA) (R = Me or ^{i}Pr; Hubert-Pfalzgraf *et al.*, 1978), $Pd_6Cu^{II}_4Cl_{12}O_4$(HMPA)$_4$ and [(HMPA)$_2$Cu^{II}Cl$_2$(PdCl$_2$)$_2$]$_n$ (Hosokawa *et al.*, 1996), and {[(PhCH$_2$)$_2$N]$_2$Mg^{II}(HMPA)}$_2$ (Clegg *et al.*, 1997). There are also morpholine complexes, such as (acetato)$_2$(morpholine)$_2$-Cu^{II}·2H$_2$O (Battaglia *et al.*, 1973), (morpholine)$_2$AlCl$_3$ (Müller & Krüger, 1984), (dmso)(morpholine)Pt^{II}Cl$_2$ (Giannikopoulos *et al.*, 2003). We present here the structure of the title compound, (I) (Fig. 1).



(I)

In (I), copper(II) is four-coordinated by one morpholine and one HMPA ligand, and two Cl atoms, to form a slightly distorted tetrahedral geometry (Fig. 1). In (I), the morpholine N—H bond is axial, because the N atom is coordinated to copper(II). However, the bond lengths and angles involving copper(II) in (I) are in good agreement with those found in other structures containing this type of group, such as those described by Mukherjee *et al.* (2005) and several other related structures. The Cu—N distance [2.0557 (12) Å] in (I) is close to previously reported values [2.050 (3) Å; Mukherjee *et al.*, 2005] and [2.064 (5) Å; Achternbosch *et al.*, 1990]. The Cu—O2 distance [1.9687 (10) Å] in (I) is also close the value reported by Mukherjee [1.965 (2) Å].

The N—H group of the morpholine ligand in (I) is engaged in an N—H···Cl hydrogen bond (Table 2). This interaction presumably contributes to the lengthening of the Cu—Cl2 bond as compared with Cu—Cl1. Compound (I) is stablized by an intramolecular C5—H5*A*···Cl1 hydrogen bond; inter-

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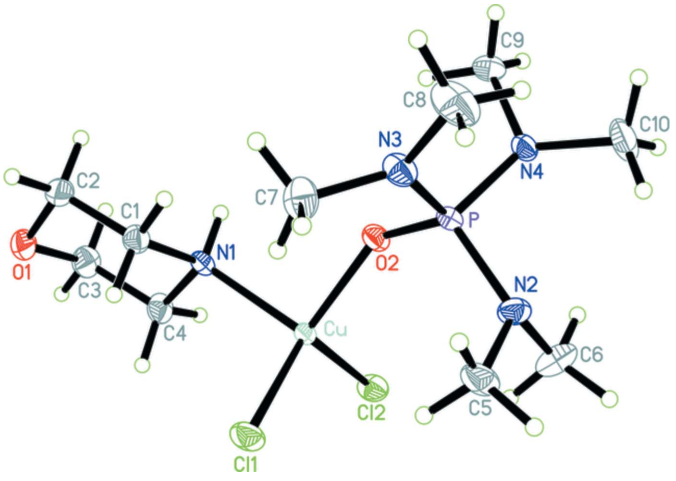

Figure 1
The molecular structure of (I) with atom labels, showing 35% probability displacement ellipsoids.

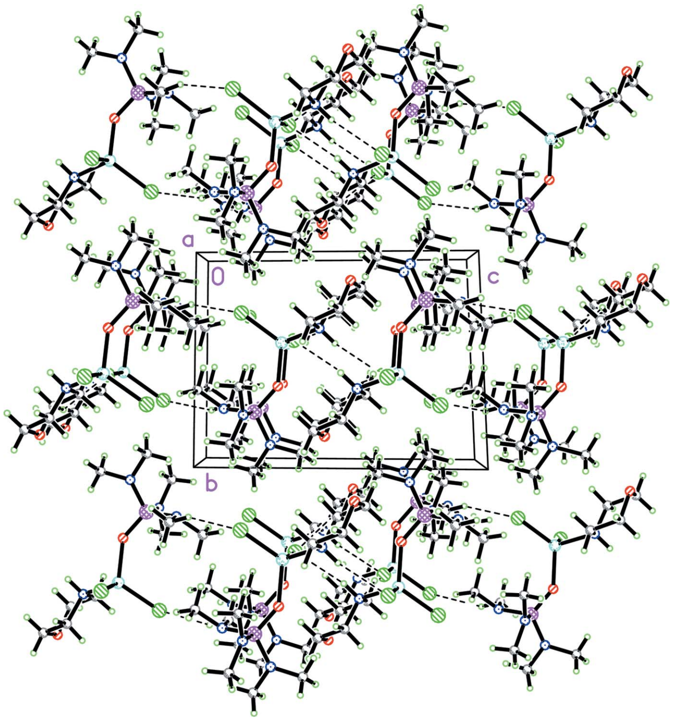

Figure 2
A packing diagram viewed along the *a* axis. Hydrogen bonds are drawn as dashed lines.

molecular hydrogen bonds are found in the crystal structure (Fig. 2 and Table 2).

Experimental

$CuCl_2$, HMPA and morpholine in a molar ratio of 1:1:1 were mixed and dissolved in sufficient water by heating to 373 K, at which point a clear solution resulted. After the reaction system was cooled slowly to room temperature, crystals of (I) were formed, collected and washed with distilled water.

Crystal data

$[CuCl_2(C_4H_9NO)(C_6H_{18}N_3OP)]$
$M_r = 400.78$
Triclinic, $P\overline{1}$
$a = 8.2649\ (3)$ Å
$b = 9.7300\ (3)$ Å
$c = 12.0927\ (3)$ Å
$\alpha = 86.150\ (1)°$
$\beta = 81.821\ (1)°$
$\gamma = 67.910\ (1)°$
$V = 891.82\ (5)$ Å^3
$Z = 2$
Mo $K\alpha$ radiation
$\mu = 1.62$ mm^{-1}
$T = 153\ (2)$ K
0.45 × 0.42 × 0.34 mm

Data collection

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*SHELXTL*; Siemens, 1998)
$T_{min} = 0.529$, $T_{max} = 0.609$ (expected range = 0.501–0.577)
8605 measured reflections
4029 independent reflections
3872 reflections with $I > 2\sigma(I)$
$R_{int} = 0.022$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.024$
$wR(F^2) = 0.063$
$S = 1.01$
4029 reflections
192 parameters
H atoms treated by a mixture of independent and constrained refinement
$\Delta\rho_{max} = 0.42$ e Å^{-3}
$\Delta\rho_{min} = -0.48$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Cu—O2	1.9687 (10)	Cu—Cl2	2.2458 (4)
Cu—N1	2.0557 (12)	P—O2	1.5032 (10)
Cu—Cl1	2.2328 (4)		
O2—Cu—N1	100.04 (4)	O2—Cu—Cl2	111.63 (3)
O2—Cu—Cl1	112.87 (3)	N1—Cu—Cl2	109.18 (3)
N1—Cu—Cl1	106.08 (3)	Cl1—Cu—Cl2	115.633 (15)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1*N*···Cl2^{i}	0.93 (2)	2.49 (2)	3.399 (2)	168 (2)
C2—H2*A*···Cl2^{ii}	0.99	2.91	3.648 (2)	132
C4—H4*A*···O1^{iii}	0.99	2.61	3.353 (2)	132
C5—H5*A*···Cl1	0.98	2.90	3.821 (2)	157
C6—H6*A*···Cl1^{iv}	0.98	2.88	3.798 (2)	156
C9—H9*C*···O1^{v}	0.98	2.94	3.915 (2)	176

Symmetry codes: (i) $-x+1, -y+1, -z+1$; (ii) $x+1, y, z$; (iii) $-x+2, -y, -z+1$; (iv) $-x+1, -y+1, -z$; (v) $x-1, y+1, z$.

The H atom of the NH group was located in a difference Fourier map and refined freely. Other H atoms were treated as riding atoms at C—H distances of 0.98–0.99 Å, with $U_{iso}(H) = 1.2U_{eq}(C)$.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2004); program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL* (Siemens, 1998); software used to prepare material for publication: *SHELXTL*.

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