metal-organic papers

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(5,5-Diphenylhydantoinato-κN³)[tris(3-aminopropyl)amine]copper(II) 5,5-diphenylhydantoinate methanol solvate

In the title compound, $[Cu(C_{15}H_{11}N_2O_2)(C_9H_{24}N_4)](C_{15}H_{11}N_2O_2) \cdot CH_3OH$, the Cu^{II} atom is coordinated in a distorted square-pyramidal coordination geometry. There are intermolecular N—H···O and O—H···N hydrogen bonds, forming a three-dimensional network.

Comment

5,5-Diphenylimidazoline-2,4-dione (phenytoin, Hpht) is a widely used drug in the treatment of epilepsy. Unfortunately, Hpht possesses toxicological properties which limit its usefulness (Milne *et al.*, 1999). We have previously reported the crystal structures of *trans*-$[Cu(pht)_2(H_2O)_2]$ (Hu, Xu, Wang *et al.*, 2006) and $[Cu(pht)_2] \cdot C_5H_{10}N_2$ (2-methyl-3,4,5,6-tetrahydropyrimidine solvate; Hu, Xu, Xu *et al.*, 2006). In this paper, we report the crystal structure of the title compound, (I).

(I)
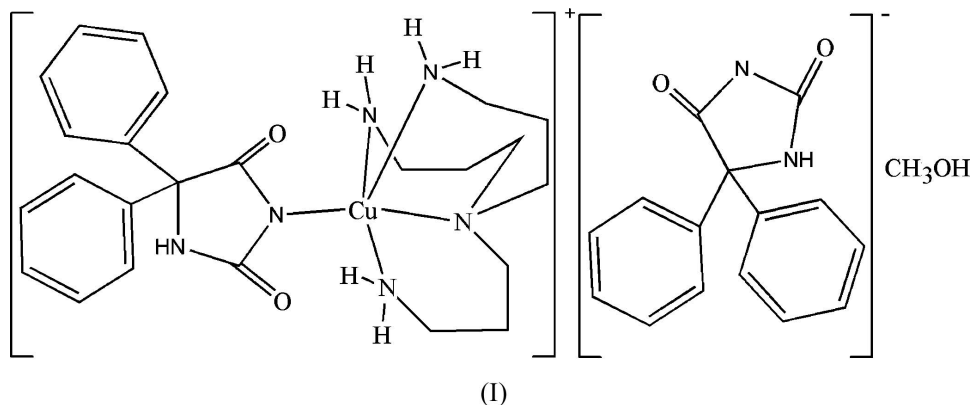
Compound (I) consists of a $[Cu(pht)(trpn)]^+$ cation [trpn = tris(3-aminopropyl)amine], a pht^- anion and a methanol solvent molecule. The Cu atom is coordinated in a distorted square-pyramidal coordination geometry by atoms N1, N7, N5 and N8 atoms in the basal plane and atom N6 at the apical position (Fig. 1 and Table 1). The dihedral angle between the plane defined by N6/N7/N8/Cu1 and the hydantoin ring of the pht ligand (N1/C1/N2/C2/C3) is 85.7 (2)°. The dihedral angles between the hydantoin ring and the phenyl groups in the pht ligand are 57.9 (2) and 74.1 (3)° for the C4–C9 and C10–C15 rings, respectively. Those angles in the pht anion are 63.5 (3) and 88.2 (3)° for the C19–C24 and C25–C30 rings, respectively. In the crystal structure, there are intermolecular N—H···O and O—H···N hydrogen bonds, forming a three-dimensional network.
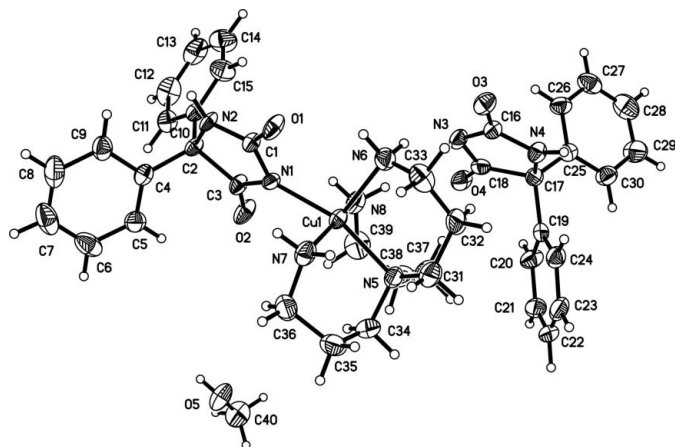
Figure 1
The asymmetric unit of (I), showing 30% probability displacement ellipsoids.

Experimental

To a solution of 5,5-diphenylhydantoin (1.00 mmol) in methanol (10 ml) was added copper(II) acetate monohydrate (0.5 mmol) and a solution of tris(3-aminopropyl)amine (1 mmol) in methanol (10 ml). The reaction mixture was stirred for 2 h at 323 K and then filtered. Blue single crystals were obtained by slow evaporation of the filtrate (yield 1.8%, m.p. 455 K). Analysis, calculated for $C_{40}H_{50}CuN_8O_5$: C 61.04, H 6.36, N 14.24%; found: C 60.89, H 6.18, N 14.31%.

Crystal data

$[Cu(C_{15}H_{11}N_2O_2)(C_9H_{24}N_4)]$-	$\gamma = 75.000\ (6)°$
$(C_{15}H_{11}N_2O_2)\cdot CH_4O$	$V = 1905.6\ (12)$ Å^3
$M_r = 786.42$	$Z = 2$
Triclinic, $P\bar{1}$	$D_x = 1.371$ Mg m^{-3}
$a = 8.562\ (3)$ Å	Mo $K\alpha$ radiation
$b = 14.548\ (5)$ Å	$\mu = 0.63$ mm^{-1}
$c = 17.323\ (7)$ Å	$T = 298\ (2)$ K
$\alpha = 66.200\ (6)°$	Block, blue
$\beta = 85.955\ (6)°$	$0.17 \times 0.12 \times 0.07$ mm

Data collection

Siemens SMART CCD area-detector diffractometer	10028 measured reflections
φ and ω scans	6630 independent reflections
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)	3034 reflections with $I > 2\sigma(I)$
$T_{min} = 0.901$, $T_{max} = 0.957$	$R_{int} = 0.066$
	$\theta_{max} = 25.0°$

Refinement

Refinement on F^2	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.075$	$w = 1/\sigma^2(F_o^2)$
$wR(F^2) = 0.252$	$(\Delta/\sigma)_{max} < 0.001$
$S = 0.99$	$\Delta\rho_{max} = 0.44$ e Å^{-3}
6630 reflections	$\Delta\rho_{min} = -0.50$ e Å^{-3}
487 parameters	

Table 1
Selected geometric parameters (Å, °).

Cu1—N8	2.019 (6)	Cu1—N5	2.086 (6)
Cu1—N1	2.023 (6)	Cu1—N6	2.174 (6)
Cu1—N7	2.030 (6)		
N8—Cu1—N1	88.5 (2)	N7—Cu1—N5	87.5 (3)
N8—Cu1—N7	163.1 (3)	N8—Cu1—N6	98.8 (3)
N1—Cu1—N7	87.7 (2)	N1—Cu1—N6	101.1 (2)
N8—Cu1—N5	91.0 (3)	N7—Cu1—N6	98.1 (3)
N1—Cu1—N5	161.8 (2)	N5—Cu1—N6	96.9 (3)

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

D—H$\cdots A$	D—H	H$\cdots A$	$D\cdots A$	D—H$\cdots A$
N2—H2$\cdots$O1^i	0.86	1.96	2.807 (7)	169
N4—H4$\cdots$O3^{ii}	0.86	2.04	2.898 (9)	173
N6—H6$A\cdots$O3	0.90	2.37	3.129 (8)	142
N6—H6$B\cdots$O1	0.90	2.27	2.975 (8)	135
N7—H7$A\cdots$O5^{iii}	0.90	2.23	3.046 (10)	151
N7—H7$B\cdots$O1	0.90	2.65	3.243 (8)	124
N8—H8$A\cdots$O2	0.90	2.55	3.056 (9)	116
N8—H8$B\cdots$N3	0.90	2.39	3.282 (9)	174
O5—H5$\cdots$N3^{iv}	0.82	1.97	2.782 (9)	170

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

All H atoms were positioned geometrically and refined in riding mode with C—H = 0.93–0.97 Å, N—H = 0.86 and 0.90 Å, and O—H = 0.82 Å, and with $U_{iso}(H)$ values of 1.2 or 1.5 times U_{eq}(parent atom).

Data collection: *SMART* (Siemens, 1996); cell refinement: *SAINT* (Siemens, 1996); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

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