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# Bis(5,5-diphenylhydantoinato-$\kappa N^3$)copper(II) 2-methyl-3,4,5,6-tetrahydropyrimidine solvate

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**Key indicators**

Single-crystal X-ray study
$T$ = 298 K
Mean $\sigma$(C–C) = 0.008 Å
Disorder in solvent or counterion
$R$ factor = 0.043
$wR$ factor = 0.104
Data-to-parameter ratio = 12.7

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



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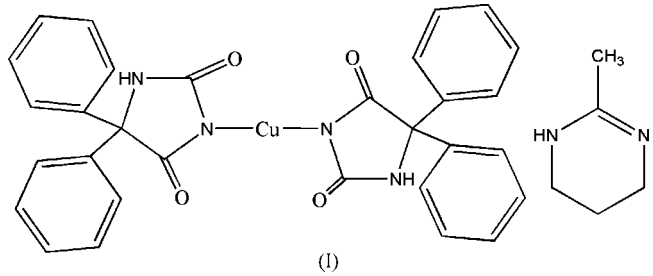
The title complex, $[Cu(C_{14}H_{11}N_2O_2)_2]\cdot C_5H_{10}N_2$, contains one $[Cu(pht)_2]$ (pht is 5,5-diphenylhydantoinate) complex molecule and one solvent molecule. The complex shows a three-dimensional network structure assembled by intermolecular N—H···O=C interactions.

## Comment

In general, copper(II) complexes exhibit a remarkable variety in their coordination geometries and numbers; moreover, gradual distortion from regular stereochemistry to distorted stereochemistries are common. 5,5-Diphenylimidazoline-2,4-dione (phenytoin, PHT) is a widely used drug in the treatment of epilepsy. Unfortunately, 5,5-diphenylimidazoline-2,4-dione possesses toxicological properties which limit its usefulness. 5,5-Diphenylimidazoline-2,4-dione should be an excellent ligand for $C^{II}$ complex formation as it contains two amide functionalities which can provide two complexible atoms (N and O) in close proximity on three faces of the heterocycle. As seen in Fig. 1, the molecular structure of the title compound, (I) contains one $[Cu(pht)_2]$ complex neutral molecule (pht is 5,5-diphenylhydantoinate) and one $C_5H_{10}N_2$ solvent molecule which results from the reaction of 1,3-propanediamine and acetic acid. The Cu atom is coordinated by two nitrogen atoms from two pht ligands. The N1—Cu1—N3 angle shows that the Cu atom exists in a distorted rectilinear geometry, with a dihedral angle of 67.7 (2)° between N1/C1/C3/N2/C2 and N3/C16/C18/N4/C17. The dihedral angle between the five-membered N3/C16/C18/N4/C17 ring and the C19–C24 phenyl ring is 64.8 (2)°, that between N1/C1/C3/N2/C2 and C4–C9 is 72.3 (2)°, that between N1/C1/C3/N2/C2 and C10–C15 is 83.1 (2)°, that between C4–C9 and C10–C15 is 71.4 (2)°, and that between C19–C24 and C25–C30 is 87.6 (2)°. The Cu—N bond distances lie in the range 1.859 (3)–1.860 (3) Å. The bond lengths and angles of (I) agree with accepted values; full details are given in the archived CIF.




(I)

From Fig. 2 it can be seen that the complex adopts a three-dimensional network structure assembled by intermolecular

N—H···O=C hydrogen bonds between the pht ligand and N—H of the heterocyclic ring.

## Experimental

To a solution of pht (1.00 mmol) in methanol (10 ml) was added copper(II) acetate monohydrate (0.5 mmol) and a solution of 1,3-propanediamine (1 mmol) in methanol (10 ml). The reaction mixture was stirred for 50 h at 423 K. Yellow crystals were obtained after cooling to room temperature (m.p. 534 K). Analysis, calculated for $C_{35}H_{32}CuN_6O_4$: C 63.23, H 4.82, N 12.65%; found: C 63.16, H 4.61, N 12.68%.

*Crystal data*

| | |
|---|---|
| $[Cu(C_{15}H_{11}N_2O_2)_2]\cdot C_5H_{10}N_2$ | $Z = 4$ |
| $M_r = 664.21$ | $D_x = 1.384$ Mg m$^{-3}$ |
| Orthorhombic, $Pna2_1$ | Mo $K\alpha$ radiation |
| $a = 22.812$ (3) Å | $\mu = 0.73$ mm$^{-1}$ |
| $b = 15.958$ (2) Å | $T = 298$ (2) K |
| $c = 8.7549$ (12) Å | Block, light-yellow |
| $V = 3187.1$ (8) Å$^3$ | 0.58 × 0.21 × 0.13 mm |

*Data collection*

| | |
|---|---|
| Bruker SMART CCD area detector diffractometer | 16303 measured reflections |
| | 5395 independent reflections |
| $\varphi$ and $\omega$ scans | 3701 reflections with $I > 2\sigma(I)$ |
| Absorption correction: multi-scan *SADABS* (Sheldrick, 1996) | $R_{int} = 0.048$ |
| | $\theta_{max} = 25.0°$ |
| $T_{min} = 0.675$, $T_{max} = 0.911$ | |

*Refinement*

| | |
|---|---|
| Refinement on $F^2$ | $w = 1/[\sigma^2(F_o^2) + (0.0398P)^2 + 0.9116P]$ |
| $R[F^2 > 2\sigma(F^2)] = 0.043$ | $(\Delta/\sigma)_{max} = 0.004$ |
| $wR(F^2) = 0.104$ | $\Delta\rho_{max} = 0.48$ e Å$^{-3}$ |
| $S = 1.03$ | $\Delta\rho_{min} = -0.25$ e Å$^{-3}$ |
| 5395 reflections | Absolute structure: Flack (1983), 2383 Friedel pairs |
| 425 parameters | |
| H-atom parameters constrained | Flack parameter: −0.035 (16) |

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

| | | | |
|---|---|---|---|
| Cu1—N1 | 1.859 (3) | Cu1—N3 | 1.860 (3) |
| N1—Cu1—N3 | 174.44 (16) | C16—N3—Cu1 | 125.5 (3) |
| C1—N1—Cu1 | 124.8 (3) | C17—N3—Cu1 | 125.1 (3) |
| C2—N1—Cu1 | 126.3 (3) | | |

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

| $D$—H···$A$ | $D$—H | H···$A$ | $D$···$A$ | $D$—H···$A$ |
|---|---|---|---|---|
| N2—H2···O4$^{i}$ | 0.86 | 1.99 | 2.828 (5) | 163 |
| N4—H4···O2$^{ii}$ | 0.86 | 2.04 | 2.902 (4) | 176 |
| N6—H6···O1 | 0.86 | 1.94 | 2.792 (5) | 169 |

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

All H atoms were positioned geometrically, with C—H = 0.93–0.97 Å and N—H = 0.86 Å, and refined using the riding-model approximation, with $U_{iso}(H) = 1.2U_{eq}(C,N)$.

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

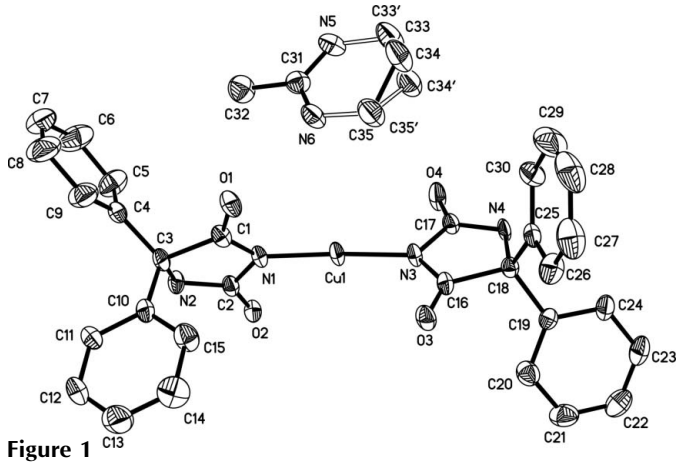



**Figure 1**
The structure of the title complex, showing 30% probability displacement ellipsoids. H atoms have been omitted. Both disorder components are shown.

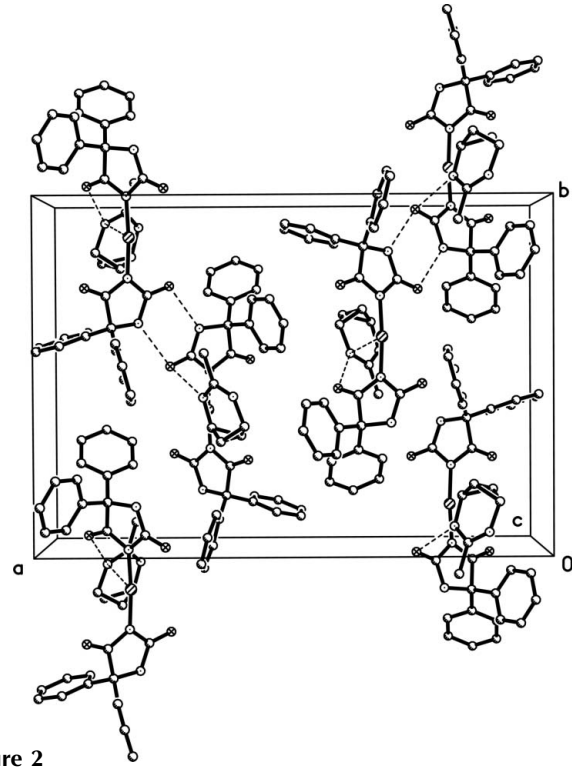



**Figure 2**
The packing of the title complex, showing the intermolecular N—H···O=C interactions (dashed lines). H atoms not involved in hydrogen bonding have been omitted.

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