

Guang-Bo Che,* Chun-Bo Liu
and Zhan-Lin XuDepartment of Chemistry, Jilin Normal
University, Siping 136000, People's Republic of
ChinaCorrespondence e-mail:
guangbochejl@yahoo.com

Key indicators

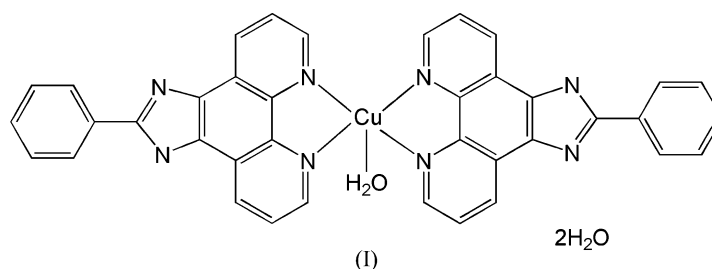
Single-crystal X-ray study
 $T = 292$ K
Mean $\sigma(\text{C}-\text{C}) = 0.007$ Å
 R factor = 0.069
 wR factor = 0.152
Data-to-parameter ratio = 15.2For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Aquabis(2-phenyl-1,3,7,8-tetraazacyclopenta-
[l]phenanthrene)copper(II) dihydrate

In the title compound, $[\text{Cu}(\text{C}_{19}\text{H}_{11}\text{N}_4)_2(\text{H}_2\text{O})] \cdot 2\text{H}_2\text{O}$ or $[\text{Cu}(\text{L})_2(\text{H}_2\text{O})] \cdot 2\text{H}_2\text{O}$, where HL is 2-phenyl-1*H*-1,3,7,8-tetraazacyclopenta[l]phenanthrene, the Cu^{II} atom is five-coordinated by four N atoms from two L^- ligands and one O atom from one water molecule. A three-dimensional supramolecular structure is formed through π - π and hydrogen-bonding interactions.

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Comment

Over the past decades, the construction of supramolecular architectures has attracted much attention from chemists because of their intriguing structural features and potential application in catalysis, magnetic devices, molecular recognition and non-linear optical materials (Abourahma *et al.*, 2002; Eddaoudi *et al.*, 2001; Lehn, 1995). It is well known that the most obvious synthetic pathway for the preparation of supramolecular frameworks is *via* direct chemical combination of functional inorganic and organic components. 1,10-Phenanthroline (phen) and its derivatives are widely employed as metal-binding components in all fields of coordination chemistry (Erkkila *et al.*, 1999). The new phen derivative 2-phenyl-1*H*-1,3,7,8-tetraazacyclopenta[l]phenanthrene (HL) possesses a multifunctional aromatic system. We report here the crystal structure of the title compound, (I), based on the L^- ligand.



In compound (I), the Cu^{II} centre is five-coordinated by four N atoms from two L^- ligands and one O atom from one water molecule, giving a slightly distorted trigonal-bipyramidal coordination, with atoms N2, N3 and O1W forming the equatorial plane and atoms N1 and N4 in axial positions (Fig. 1).

The Cu–N bond lengths vary from 1.986 (3) to 2.129 (4) Å and the Cu–O1W distance is 2.051 (4) Å. It should be noted that the N atoms of the imidazole rings of the ligands balance the charge of Cu^{2+} , generating a neutral complex.

In the crystal packing, centrosymmetrically related L^- ligands [N3/N4/N7/N8/C20–C38 at (x, y, z) and $(1 - x, 1 - y, 1 - z)$] are linked into dimers by π - π interactions (the

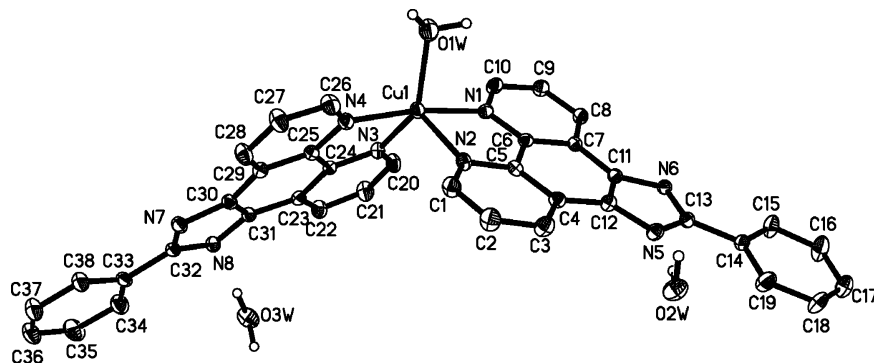


Figure 1

The asymmetric unit of compound (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. C-bound H atoms have been omitted.

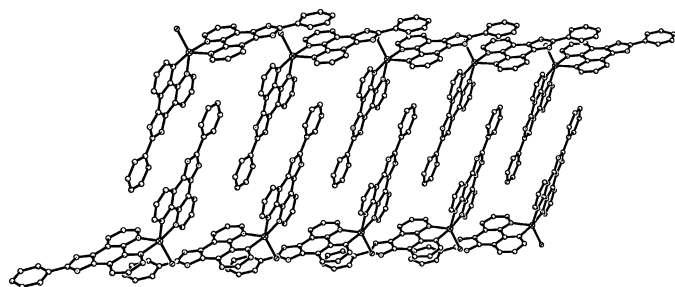


Figure 2

View of the ladder-like supramolecular structure of (I). H atoms have been omitted for clarity.

distance between the mean planes of the ligands is 3.59 Å) and O—H...N hydrogen-bond interactions (Table 1), resulting in a ladder-like supramolecular structure (Fig. 2).

Experimental

The HL ligand was synthesized according to the reported procedure of Steck & Day (1943). Compound (I) was hydrothermally synthesized under autogenous pressure. A mixture of HL, CuSO₄ and water in a 2:1:5000 molar ratio was stirred and the pH was adjusted to about 9 with ammonia. The system was then sealed in a Teflon-lined autoclave and heated at 423 K for 3 d. Blue block-shaped crystals were obtained by slow cooling of the reaction mixture (yield 71% based on Cu).

Crystal data

[Mn(C₁₉H₁₁N₄)₂(H₂O)]·2H₂O
M_r = 708.22
 Monoclinic, *P*2₁/*c*
a = 9.2897 (19) Å
b = 11.466 (2) Å
c = 30.189 (6) Å
 β = 95.85 (3)°
V = 3198.9 (11) Å³

Z = 4
D_x = 1.471 Mg m⁻³
 Mo *K*α radiation
 μ = 0.74 mm⁻¹
T = 292 (2) K
 Block, blue
 0.33 × 0.26 × 0.18 mm

Data collection

Rigaku R-Axis RAPID
 diffractometer
 ω scans
 Absorption correction: multi-scan
 (ABSCOR; Higashi, 1995)
T_{min} = 0.777, *T_{max}* = 0.875

29006 measured reflections
 7231 independent reflections
 3942 reflections with *I* > 2σ(*I*)
R_{int} = 0.113
 θ_{\max} = 27.5°

Refinement

Refinement on *F*²
R[*F*² > 2σ(*F*²)] = 0.069
wR(*F*²) = 0.152
S = 1.02
 7231 reflections
 475 parameters
 H atoms treated by a mixture of
 independent and constrained
 refinement

$$w = 1/[\sigma^2(F_o^2) + (0.0471P)^2 + 3.7579P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$
 $\Delta\rho_{\max} = 0.96 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.44 \text{ e \AA}^{-3}$

Table 1

Hydrogen-bond geometry (Å, °).

<i>D</i> —H... <i>A</i>	<i>D</i> —H	H... <i>A</i>	<i>D</i> ... <i>A</i>	<i>D</i> —H... <i>A</i>
O1W—HW11...N6 ⁱ	0.94 (2)	2.39 (3)	3.292 (5)	162 (6)
O1W—HW12...O2W ⁱⁱ	0.94 (2)	2.12 (2)	3.050 (5)	172 (5)
O2W—HW21...N5	0.84 (7)	1.98 (7)	2.819 (5)	171 (7)
O3W—HW31...N8	0.94 (7)	1.91 (7)	2.849 (6)	177 (6)
O3W—HW32...N7 ⁱⁱⁱ	0.85 (8)	2.16 (7)	2.871 (5)	141 (7)

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

All H atoms attached to C atoms were positioned geometrically and refined as riding atoms, with C—H = 0.93 Å and *U*_{iso}(H) = 1.2*U*_{eq}(C). The water H atoms were located in difference Fourier maps and refined freely. The high *R*_{int} value of 0.113 is the result of weak high-angle data.

Data collection: *PROCESS-AUTO* (Rigaku, 1998); cell refinement: *PROCESS-AUTO*; data reduction: *PROCESS-AUTO*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *SHELXTL-Plus* (Sheldrick, 1990); software used to prepare material for publication: *SHELXL97*.

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