

**2',7'-Di-4-pyridylspiro[cyclopropane-1,9'-fluorene]
hemihydrate****Zi-Xing Wang, Hai-Yao Lin and
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Key indicatorsSingle-crystal X-ray study
 $T = 296$ K
Mean $\sigma(\text{C}-\text{C}) = 0.002$ Å
 R factor = 0.040
 wR factor = 0.072
Data-to-parameter ratio = 16.7For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.

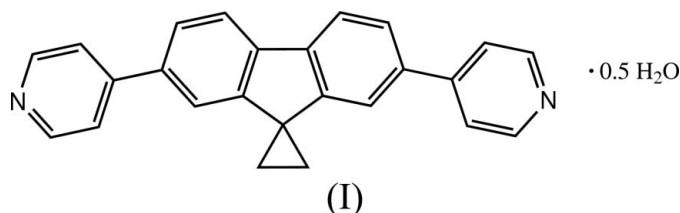
The asymmetric unit of the crystal structure of the title compound, $\text{C}_{25}\text{H}_{18}\text{N}_2 \cdot 0.5\text{H}_2\text{O}$, contains two independent dipyridylspiro(cyclopropanefluorene) (CPF) molecules and one solvent water molecule. The two CPF molecules show significantly different dihedral angles between the pyridine rings and fluorene plane. The water molecule links with the CPF molecules *via* $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonding to form a one-dimensional supramolecular chain.

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Comment

Lewis base receptor sites and shape-selective cavities have been incorporated within coordination-directed supramolecular ensembles. A ligand containing two pyridyl rings is one of the most popular methods for designing such structures (Yamamoto *et al.* 2003). The title compound, (I), was synthesized by the Suzuki reaction. The cyclopropane ring, as the smallest dialkyl group, is introduced at the C-9 position of fluorene.



Two independent dipyridylspiro(cyclopropanefluorene) (CPF) molecules and one solvent water molecule occur in the asymmetric unit (Fig. 1). The two independent CPF molecules have similar bond lengths (Table 1), but the conformations in the two CPF molecules are significantly different. In the C1-containing molecule, the N11-pyridine and N12-pyridine rings are twisted with respect to the mean plane of the fluorene system with dihedral angles of $34.91(5)^\circ$ and $29.43(5)^\circ$, respectively. In the C31-containing molecule, the N31-pyridine and N32-pyridine rings are twisted similarly by $23.26(6)^\circ$ and $29.23(6)^\circ$, respectively.

The water molecule links with the CPF molecules *via* $\text{O}-\text{H} \cdots \text{N}$ hydrogen bonding to form a one-dimensional supramolecular chain (Table 2).

Experimental

Compound (I) was prepared by a method similar to that reported previously (Wang *et al.*, 2006). The crude products were purified by column chromatography (silica gel) using ethyl acetate as eluant. In this way, (I) was obtained as a pale-yellow solid in 70% yield. Single

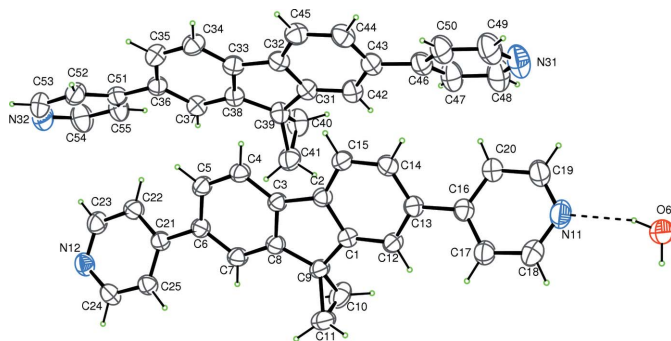


Figure 1
The asymmetric unit of (I), with 50% probability displacement ellipsoids (arbitrary spheres for H atoms). The dashed line indicates hydrogen bonding.

crystals of (I) were obtained by slow evaporation of an ethyl acetate solution at room temperature.

Crystal data

$C_{25}H_{18}N_2 \cdot H_2O$
 $M_r = 355.44$
Monoclinic, $P2_1/c$
 $a = 14.883$ (3) Å
 $b = 22.675$ (5) Å
 $c = 11.334$ (2) Å
 $\beta = 105.817$ (9)°
 $V = 3680.1$ (13) Å³

$Z = 8$
 $D_x = 1.283$ Mg m⁻³
Mo $K\alpha$ radiation
 $\mu = 0.08$ mm⁻¹
 $T = 296$ (1) K
Block, yellow
0.32 × 0.28 × 0.17 mm

Data collection

Rigaku R-Axis RAPID
diffractometer
 ω scans
Absorption correction: none
34544 measured reflections

8316 independent reflections
4200 reflections with $F^2 > 2\sigma(F^2)$
 $R_{int} = 0.041$
 $\theta_{max} = 27.5^\circ$

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.040$
 $wR(F^2) = 0.072$
 $S = 1.00$
8316 reflections
497 parameters

H-atom parameters constrained
 $w = 1/[\sigma(F_o^2)]/(4F_o^2)$
 $(\Delta/\sigma)_{max} < 0.001$
 $\Delta\rho_{max} = 0.33$ e Å⁻³
 $\Delta\rho_{min} = -0.29$ e Å⁻³

Table 1
Selected bond lengths (Å).

C9—C10	1.5030 (19)	C39—C40	1.5142 (19)
C9—C11	1.507 (2)	C39—C41	1.510 (2)
C10—C11	1.469 (2)	C40—C41	1.475 (2)

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

$D-H \cdots A$	$D-H$	$H \cdots A$	$D \cdots A$	$D-H \cdots A$
O6—H601 ⁱ ···N11	0.87	2.04	2.872 (2)	160
O6—H602 ⁱ ···N12 ⁱ	0.85	2.05	2.8928 (17)	170

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

Water H atoms were located in a difference Fourier map and refined as riding in their as-found relative positions, with $U_{iso}(H) = 1.2U_{eq}(O)$. Other H atoms were placed in calculated positions with $C-H = 0.93$ Å (aromatic) or 0.97 Å (methylene) and refined in riding mode, $U_{iso}(H) = 1.2U_{eq}(C)$.

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

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References

- Rigaku (1998). *PROCESS-AUTO*. Rigaku Corporation, Tokyo, Japan.
Rigaku/MS (2004). *CrystalStructure*. Version 3.7.0. Rigaku/MS, The Woodlands, Texas, USA.
Sheldrick, G. M. (1997). *SHELXS97* and *SHELXL97*. University of Göttingen, Germany.
Wang, Z.-X., Lin, H.-Y. & Lu, P. (2006). *Acta Cryst.* **E62**, o1084–o1085.
Yamamoto, T., Arif, A. M. & Stang, P. J. (2003). *J. Am. Chem. Soc.* **125**, 12309–12317.