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The crystal structure of $N^6, N^{6'}$ -di(pyridin-2-yl)-[2,2'-bipyridine]-6,6'-diamine, $C_{20}H_{16}N_6$

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Abstract

$C_{20}H_{16}N_6$, monoclinic, $P2_1/c$ (no. 14), $a = 7.6623(4)$ Å, $b = 11.8206(5)$ Å, $c = 8.8960(4)$ Å, $\beta = 91.825(2)^\circ$, $V = 805.33(7)$ Å³, $Z = 2$, $R_{gt}(F) = 0.0577$, $wR_{ref}(F^2) = 0.1588$, $T = 200(2)$ K.

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Table 1 contains crystallographic data and Table 2 contains the list of the atoms including atomic coordinates and displacement parameters.

Table 1: Data collection and handling.

Crystal:	Prism, colorless
Size:	$0.35 \times 0.18 \times 0.16$ mm
Wavelength:	Mo $K\alpha$ radiation (0.71073 Å)
μ :	0.09 mm ⁻¹
Diffractometer, scan mode:	SCXmini, ω -scans
θ_{max} , completeness:	27.5°, >99%
$N(hkl)_{measured}$, $N(hkl)_{unique}$, R_{int} :	11,399, 1848, 0.042
Criterion for I_{obs} , $N(hkl)_{gt}$:	$I_{obs} > 2 \sigma(I_{obs})$, 1504
$N(param)_{refined}$:	119
Programs:	CrystalClear [1], OLEX2 [2], SHELX [3, 4]

Source of materials

About 2.0 mmol 6,6'-diamino-2,2'-bipyridine, 6.0 mmol 2-bromopyridine and 8.1 mmol KO^tBu were weighed in a dry Schlenk tube, 0.040 mmol Pd₂(dba)₃, 0.08 mmol

BINAP as catalyst and 30 mL of dry, degassed toluene as solvent were added. The mixture was heated to 80 °C and stirred under argon for three days before it was cooled to room temperature. After that 150 mL water were added and stirred vigorously for 1 h. The solid was filtered out and the title product was obtained by recrystallization from a hot mixture of methanol and DMF.

Experimental details

Hydrogen atoms were placed in their geometrically idealized positions and constrained to ride on their parent atoms.

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	U _{iso} [*] /U _{eq}
N2	0.17893 (18)	0.90166 (11)	0.49443 (14)	0.0247 (4)
N1	0.3891 (2)	0.76545 (13)	0.49976 (16)	0.0316 (4)
H1	0.422127	0.799670	0.417555	0.038*
N3	0.4950 (2)	0.64946 (12)	0.69759 (16)	0.0303 (4)
C1	0.0235 (2)	0.94368 (13)	0.53295 (17)	0.0234 (4)
C10	0.2317 (2)	0.80296 (14)	0.55461 (17)	0.0253 (4)
C4	0.1321 (2)	0.74258 (14)	0.65720 (19)	0.0291 (4)
H4	0.173922	0.674092	0.701095	0.035*
C2	−0.0862 (2)	0.88787 (15)	0.62940 (19)	0.0293 (4)
H2	−0.197151	0.918115	0.652035	0.035*
C5	0.5019 (2)	0.68280 (14)	0.55401 (18)	0.0265 (4)
C3	−0.0283 (2)	0.78606 (15)	0.6920 (2)	0.0314 (4)
H3	−0.100128	0.746299	0.759236	0.038*
C6	0.6245 (2)	0.63903 (15)	0.4550 (2)	0.0311 (4)
H6	0.628163	0.665418	0.354263	0.037*
C7	0.7385 (3)	0.55759 (16)	0.5067 (2)	0.0346 (5)
H7	0.822917	0.526926	0.442220	0.042*
C8	0.7297 (3)	0.52024 (16)	0.6542 (2)	0.0356 (5)
H8	0.806287	0.463001	0.691977	0.043*
C9	0.6076 (3)	0.56813 (16)	0.7439 (2)	0.0348 (5)
H9	0.602091	0.542426	0.844852	0.042*

Comment

The title compound is an useful organic blocks for the synthesis of metal organic hybrid phase transition materials [5–7], which are used in nonvolatile memory storage [8], electronics [9], optics [10] and telecom shelters [11]. It is also an interesting work to try to detect metal ion pollutants using this compound [12]. In our exploration on multiferroic compounds and searching for new metal organic hybrid phase transition materials [13], we come across the crystal structure of the title compound. In the title molecule, all bond lengths are in normal ranges [14–16]. The molecular structure is centro-symmetric arranged around a inversion center (see the Figure). The dihedral angle between the pyridine rings of each half molecular is 33.8°. In the crystal, N–H···N hydrogen bonds stabilized the structure.

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