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2,2′,6,6′-Tetramethyl-4,4′-bipyridine

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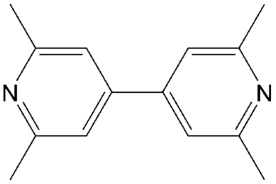
Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.002 Å; R factor = 0.051; wR factor = 0.164; data-to-parameter ratio = 19.9.

In the title compound, $C_{14}H_{16}N_2$, which has no crystallographic molecular symmetry, the dihedral angle between the least-squares planes of the two pyridine rings is 19.48 (2)°. No classical hydrogen bonds nor π–π interactions were found.

Related literature

For the synthesis of the title compound, see: Hunig & Wehner (1989). For related compounds, see: Coles *et al.* (2002); Jackisch *et al.* (1990); Lin *et al.* (2006).



Experimental

Crystal data

$C_{14}H_{16}N_2$	a = 21.9827 (10) Å
M_r = 212.29	c = 10.1569 (6) Å
Tetragonal, $I4_1/a$	V = 4908.2 (4) Å^3
Z = 16	T = 293 (2) K
Mo $K\alpha$ radiation	0.32 × 0.26 × 0.26 mm
μ = 0.07 mm^{-1}	

Data collection

Bruker SMART APEXII diffractometer	14604 measured reflections
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)	2959 independent reflections
$T_{\min}$ = 0.979, $T_{\max}$ = 0.982	2112 reflections with $I > 2\sigma(I)$
	$R_{\rm int}$ = 0.031

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.051	149 parameters
$wR(F^2)$ = 0.165	H-atom parameters constrained
S = 1.06	$\Delta\rho_{\max}$ = 0.22 e Å^{-3}
2959 reflections	$\Delta\rho_{\min}$ = −0.18 e Å^{-3}

Table 1
Selected torsion angles (°).

C2—C3—C8—C7	−20.9 (2)	C2—C3—C8—C9	159.45 (14)
C4—C3—C8—C7	159.85 (14)	C4—C3—C8—C9	−19.8 (2)

Data collection: *APEX2* (Bruker, 2004); cell refinement: *SAINT* (Bruker, 2004); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2001); program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL* and local programs.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: OM2191).

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supporting information

Acta Cryst. (2008). E**64**, o127 [https://doi.org/10.1107/S1600536807063040]

2,2′,6,6′-Tetramethyl-4,4′-bipyridine

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S1. Comment

The title compound, $C_{14}H_{16}N_2$, is a important synthetic intermediate for the preparation of the ligand 4,4′-bipyridine-2,6,2′,6′-tetracarboxylic acid, which can be used for constructing open and robust coordination frameworks (Lin *et al.*, 2006).

The molecular structure of the compound is shown in Fig. 1, and selected geometric parameters are given in Table 1. The dihedral angle between the least-squares planes of the two pyridine rings is 19.48 (2) °. This is probably because of steric hindrance, which is similar to related compounds (Coles *et al.*, 2002, Jackisch *et al.*, 1990, Lin *et al.*, 2006), and for this reason the molecule has no symmetry plane.

In the crystal structure no classic hydrogen bonds nor π-π interactions were found (Fig. 2). The molecules may be linked together by weak van der Waals interactions.

S2. Experimental

All reagents were purchased from Aldrich and used without further purification. The compound was synthesized according to a reported method (Hunig & Wehner, 1989). It (0.424 g, 0.002 mol) was dissolved in ethanol (20 ml). After heating at 343 K for 20 min, the mixture was allowed to cool and evaporate naturally. After a few days, yellow crystalline lumps formed. Analysis found: C 79.24, H 7.60, N 13.16%.; $C_{14}H_{16}N_2$ requires: C 79.20, H 7.60, N 13.20%.

S3. Refinement

All H atoms were positioned geometrically and refined as riding atoms with C—H distances = 0.93–0.97 A °. For the aromatic H atoms U_{iso}(H) = 1.2Ueq(C), and for the CH3 H atoms U_{iso}(H) = 1.5Ueq(C). The highest peak 0.22 e.A^-3^ in the final difference map is located 0.81 Å from H11C.

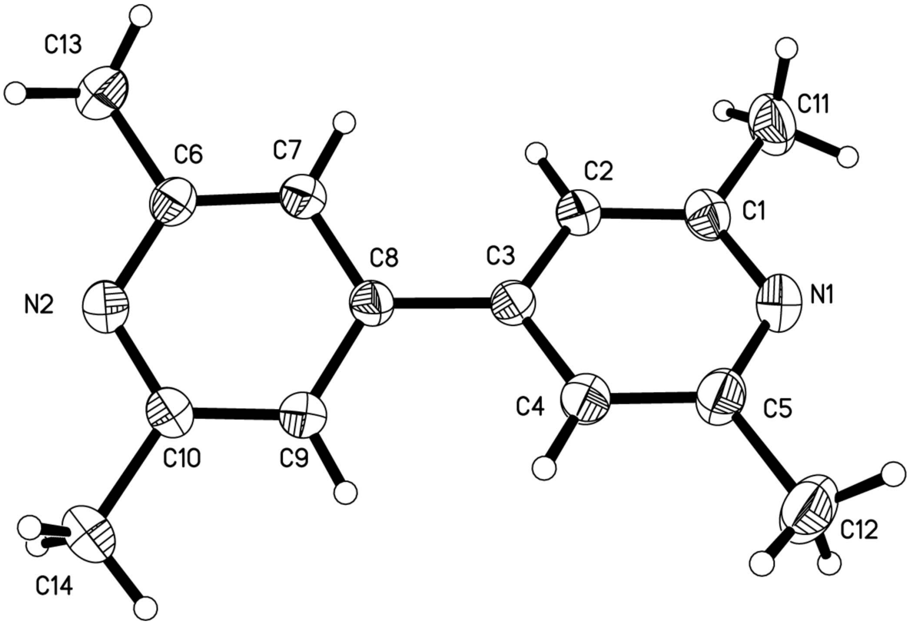

Figure 1

The molecular structure showing the atom-labeling scheme, with displacement ellipsoids drawn at the 30% probability level.

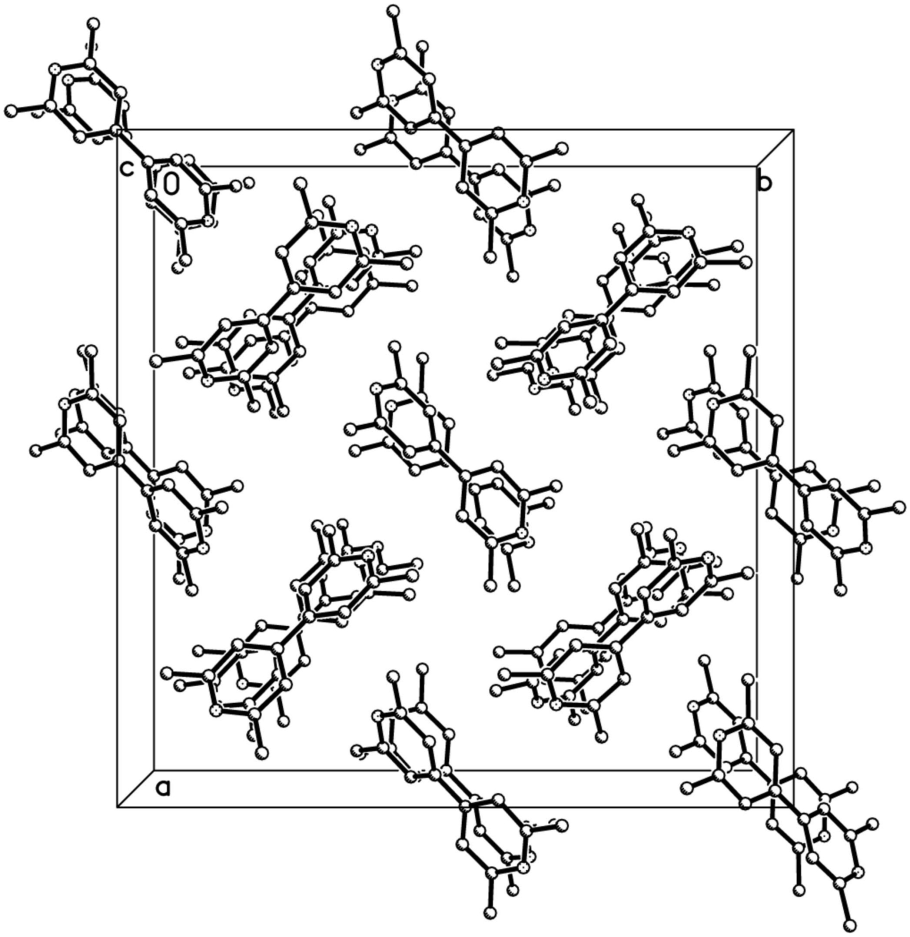

Figure 2

The crystal packing, viewed along the *c* axis.

2,2′,6,6′-Tetramethyl-4,4′-bipyridine

Crystal data

$C_{14}H_{16}N_2$
$M_r = 212.29$
Tetragonal, $I4_1/a$
$a = 21.9827\ (10)$ Å
$c = 10.1569\ (6)$ Å
$V = 4908.2\ (4)$ Å^3
$Z = 16$
$F(000) = 1824$
$D_x = 1.149$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 352 reflections
$\theta = 2.6$–$24.8°$
$\mu = 0.07$ mm^{-1}
$T = 293$ K
Block, yellow
0.32 × 0.26 × 0.26 mm

Data collection

Bruker SMART APEXII diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2004)
$T_{min} = 0.979$, $T_{max} = 0.982$
14604 measured reflections
2959 independent reflections
2112 reflections with $I > 2\sigma(I)$
$R_{int} = 0.031$
$\theta_{max} = 28.3°$, $\theta_{min} = 1.9°$
$h = -28 \rightarrow 28$
$k = -21 \rightarrow 29$
$l = -12 \rightarrow 13$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.051$
$wR(F^2) = 0.165$
$S = 1.06$
2959 reflections
149 parameters
0 restraints
Primary atom site location: structure-invariant direct methods
Secondary atom site location: difference Fourier map
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0889P)^2 + 1.2656P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.002$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.18$ e Å^{-3}

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R-factor wR and goodness of fit S are based on F^2, conventional R-factors R are based on F, with F set to zero for negative F^2. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. R-factors based on F^2 are statistically about twice as large as those based on F, and R- factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å^2)

	x	y	z	U_{iso}*/U_{eq}
N1	0.36926 (6)	0.13022 (6)	−0.07996 (14)	0.0547 (4)
N2	0.14766 (6)	0.34910 (6)	0.10403 (13)	0.0501 (3)
C1	0.33348 (7)	0.12075 (7)	0.02437 (16)	0.0486 (4)
C2	0.28970 (6)	0.16252 (6)	0.06394 (15)	0.0442 (3)
H2	0.2659	0.1545	0.1377	0.053*
C3	0.28130 (6)	0.21610 (6)	−0.00610 (14)	0.0415 (3)
C4	0.31887 (7)	0.22525 (7)	−0.11452 (16)	0.0509 (4)
H4	0.3149	0.2604	−0.1649	0.061*
C5	0.36228 (7)	0.18207 (8)	−0.14767 (16)	0.0551 (4)
C6	0.14380 (6)	0.29160 (7)	0.14839 (15)	0.0453 (4)
C7	0.18561 (6)	0.24730 (7)	0.11270 (15)	0.0449 (4)
H7	0.1805	0.2075	0.1420	0.054*
C8	0.23495 (6)	0.26192 (6)	0.03364 (14)	0.0411 (3)
C9	0.23940 (7)	0.32206 (6)	−0.00853 (16)	0.0483 (4)
H9	0.2722	0.3343	−0.0600	0.058*
C10	0.19490 (7)	0.36346 (6)	0.02641 (16)	0.0504 (4)
C11	0.34380 (10)	0.06300 (8)	0.1000 (2)	0.0704 (5)
H11A	0.3147	0.0330	0.0727	0.106*
H11B	0.3391	0.0708	0.1924	0.106*
H11C	0.3842	0.0483	0.0832	0.106*
C12	0.40499 (10)	0.19262 (11)	−0.2612 (2)	0.0815 (6)
H12A	0.4440	0.2051	−0.2282	0.122*
H12B	0.3888	0.2238	−0.3173	0.122*
H12C	0.4094	0.1556	−0.3106	0.122*
C13	0.09245 (7)	0.27763 (9)	0.24106 (17)	0.0588 (4)

H13A	0.1058	0.2836	0.3301	0.088*
H13B	0.0799	0.2361	0.2295	0.088*
H13C	0.0588	0.3042	0.2228	0.088*
C14	0.19686 (10)	0.42776 (8)	−0.0243 (2)	0.0759 (6)
H14A	0.1633	0.4344	−0.0832	0.114*
H14B	0.2344	0.4344	−0.0703	0.114*
H14C	0.1940	0.4556	0.0484	0.114*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0522 (7)	0.0543 (8)	0.0574 (8)	0.0114 (6)	0.0035 (6)	−0.0052 (6)
N2	0.0475 (7)	0.0470 (7)	0.0558 (8)	0.0060 (5)	0.0012 (6)	−0.0011 (6)
C1	0.0492 (8)	0.0439 (8)	0.0527 (9)	0.0059 (6)	−0.0042 (7)	−0.0017 (6)
C2	0.0426 (7)	0.0413 (7)	0.0487 (8)	0.0012 (6)	0.0013 (6)	0.0010 (6)
C3	0.0363 (7)	0.0388 (7)	0.0495 (8)	−0.0006 (5)	−0.0014 (6)	−0.0011 (6)
C4	0.0512 (8)	0.0480 (8)	0.0537 (9)	0.0045 (6)	0.0070 (7)	0.0061 (7)
C5	0.0518 (9)	0.0597 (10)	0.0539 (9)	0.0059 (7)	0.0080 (7)	−0.0024 (7)
C6	0.0385 (7)	0.0505 (8)	0.0469 (8)	0.0001 (6)	−0.0023 (6)	−0.0008 (6)
C7	0.0427 (7)	0.0400 (7)	0.0519 (8)	−0.0006 (6)	0.0013 (6)	0.0047 (6)
C8	0.0384 (7)	0.0380 (7)	0.0470 (8)	0.0010 (5)	−0.0017 (6)	0.0004 (6)
C9	0.0446 (8)	0.0423 (8)	0.0579 (9)	0.0005 (6)	0.0063 (7)	0.0056 (6)
C10	0.0503 (8)	0.0403 (8)	0.0606 (9)	0.0038 (6)	0.0010 (7)	0.0022 (7)
C11	0.0815 (13)	0.0534 (10)	0.0764 (13)	0.0223 (9)	0.0073 (10)	0.0065 (9)
C12	0.0752 (13)	0.0951 (15)	0.0743 (13)	0.0159 (11)	0.0300 (10)	0.0069 (11)
C13	0.0466 (9)	0.0706 (11)	0.0590 (10)	0.0001 (7)	0.0070 (7)	0.0017 (8)
C14	0.0772 (13)	0.0447 (9)	0.1057 (16)	0.0116 (8)	0.0158 (11)	0.0139 (9)

Geometric parameters (Å, º)

N1—C1	1.336 (2)	C8—C9	1.3931 (19)
N1—C5	1.340 (2)	C9—C10	1.383 (2)
N2—C10	1.341 (2)	C9—H9	0.9300
N2—C6	1.3446 (19)	C10—C14	1.505 (2)
C1—C2	1.389 (2)	C11—H11A	0.9600
C1—C11	1.501 (2)	C11—H11B	0.9600
C2—C3	1.3885 (19)	C11—H11C	0.9600
C2—H2	0.9300	C12—H12A	0.9600
C3—C4	1.391 (2)	C12—H12B	0.9600
C3—C8	1.4886 (19)	C12—H12C	0.9600
C4—C5	1.388 (2)	C13—H13A	0.9600
C4—H4	0.9300	C13—H13B	0.9600
C5—C12	1.505 (2)	C13—H13C	0.9600
C6—C7	1.387 (2)	C14—H14A	0.9600
C6—C13	1.502 (2)	C14—H14B	0.9600
C7—C8	1.3873 (19)	C14—H14C	0.9600
C7—H7	0.9300		

C1—N1—C5	118.18 (13)	C8—C9—H9	120.1
C10—N2—C6	117.84 (12)	N2—C10—C9	122.93 (13)
N1—C1—C2	122.29 (14)	N2—C10—C14	116.37 (14)
N1—C1—C11	116.65 (14)	C9—C10—C14	120.69 (15)
C2—C1—C11	121.04 (15)	C1—C11—H11A	109.5
C3—C2—C1	120.29 (14)	C1—C11—H11B	109.5
C3—C2—H2	119.9	H11A—C11—H11B	109.5
C1—C2—H2	119.9	C1—C11—H11C	109.5
C2—C3—C4	116.71 (13)	H11A—C11—H11C	109.5
C2—C3—C8	121.74 (13)	H11B—C11—H11C	109.5
C4—C3—C8	121.55 (13)	C5—C12—H12A	109.5
C5—C4—C3	120.10 (14)	C5—C12—H12B	109.5
C5—C4—H4	120.0	H12A—C12—H12B	109.5
C3—C4—H4	120.0	C5—C12—H12C	109.5
N1—C5—C4	122.42 (15)	H12A—C12—H12C	109.5
N1—C5—C12	116.94 (15)	H12B—C12—H12C	109.5
C4—C5—C12	120.63 (16)	C6—C13—H13A	109.5
N2—C6—C7	122.05 (13)	C6—C13—H13B	109.5
N2—C6—C13	116.73 (13)	H13A—C13—H13B	109.5
C7—C6—C13	121.21 (14)	C6—C13—H13C	109.5
C6—C7—C8	120.44 (13)	H13A—C13—H13C	109.5
C6—C7—H7	119.8	H13B—C13—H13C	109.5
C8—C7—H7	119.8	C10—C14—H14A	109.5
C7—C8—C9	116.92 (13)	C10—C14—H14B	109.5
C7—C8—C3	122.38 (12)	H14A—C14—H14B	109.5
C9—C8—C3	120.70 (13)	C10—C14—H14C	109.5
C10—C9—C8	119.74 (13)	H14A—C14—H14C	109.5
C10—C9—H9	120.1	H14B—C14—H14C	109.5

C5—N1—C1—C2	0.2 (2)	N2—C6—C7—C8	2.9 (2)
C5—N1—C1—C11	−178.33 (16)	C13—C6—C7—C8	−175.91 (14)
N1—C1—C2—C3	0.7 (2)	C6—C7—C8—C9	−1.0 (2)
C11—C1—C2—C3	179.13 (15)	C6—C7—C8—C3	179.37 (13)
C1—C2—C3—C4	−0.6 (2)	C2—C3—C8—C7	−20.9 (2)
C1—C2—C3—C8	−179.86 (13)	C4—C3—C8—C7	159.85 (14)
C2—C3—C4—C5	−0.3 (2)	C2—C3—C8—C9	159.45 (14)
C8—C3—C4—C5	178.97 (14)	C4—C3—C8—C9	−19.8 (2)
C1—N1—C5—C4	−1.1 (2)	C7—C8—C9—C10	−1.5 (2)
C1—N1—C5—C12	177.52 (16)	C3—C8—C9—C10	178.14 (14)
C3—C4—C5—N1	1.2 (3)	C6—N2—C10—C9	−0.4 (2)
C3—C4—C5—C12	−177.38 (16)	C6—N2—C10—C14	178.45 (16)
C10—N2—C6—C7	−2.2 (2)	C8—C9—C10—N2	2.3 (3)
C10—N2—C6—C13	176.71 (14)	C8—C9—C10—C14	−176.52 (16)