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3,5-Dimethyl-1,7-diphenyl-4-(2-pyridyl)-1*H*,7*H*-pyrazolo[3,4-*b*:4′,3′-e]pyridine

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Key indicators

Single-crystal X-ray study
T = 120 K
Mean σ(C–C) = 0.002 Å
R factor = 0.040
wR factor = 0.110
Data-to-parameter ratio = 15.9

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

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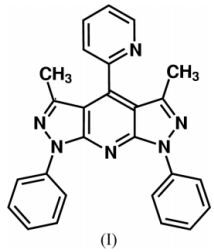
In the title compound, $C_{26}H_{20}N_6$, the supramolecular structure is determined by a weak C—H···N hydrogen bond which links the molecules into chains running parallel to [110].

Comment

In our ongoing application of microwave irradiation to heterocyclic synthesis by multicomponent condensation, we have prepared compound (I) from 5-amino-3-methyl-1-phenyl-1*H*-pyrazole and 2-pyridinecarbadehyde.



(I)

There are no unusual bonds and angles in the structure (Table 1 and Fig. 1). The 12-membered fused-ring system defined by atoms N1 to C8*A* is essentially planar and symmetrical about the N1···C5 line, as shown by the bonds and angles in Table 1. However, the angle of tilt of the phenyl rings out of the plane of the 12-membered ring system varies as is shown by the torsion angles listed in Table 1 so that this

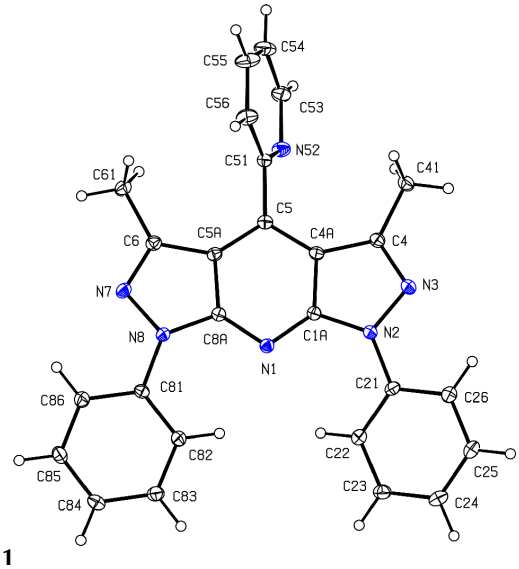


Figure 1
A view of (I), with the atomic numbering scheme. Displacement ellipsoids are drawn at the 30% probability level.

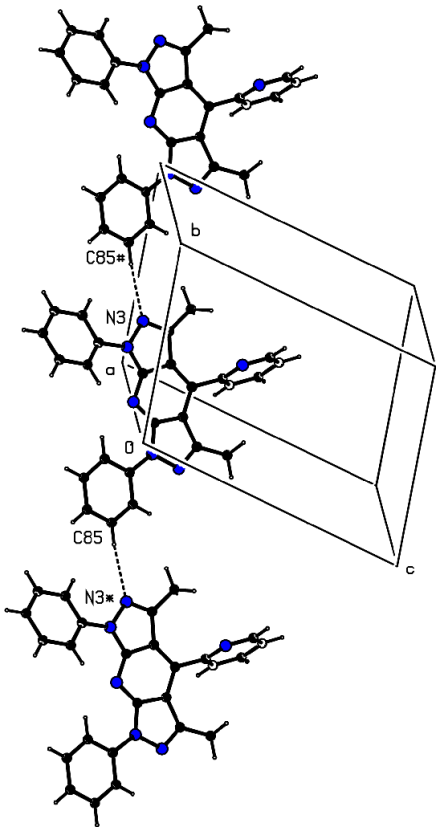


Figure 2
A view of the *C*(10) chains running parallel to [110]. The atom labelled with an asterisk (*) is in the molecule at $(x-1, y-1, z)$ while that labelled with a hash (#) is in the molecule at $(1+x, 1+y, z)$.

symmetry does not extend to the whole molecule. There are two short intramolecular contacts C22—H22···N1 and C82—H82···N1, *i.e.* between the two phenyl rings defined by atoms C21–C26 and C81–C86 to atom N1 (Table 2). The C8—H85···N3 weak hydrogen bond links the molecules into *C*(10) chains (Bernstein *et al.*, 1995) which run parallel to [110] (Fig. 2).

Experimental

A mixture of 5-amino-3-methyl-1-phenylpyrazole (10 mmol) and 2-pyridinecarbaldehyde (20 mmol) was placed into Pyrex-glass open vessels and irradiated in a domestic microwave oven for 1.5 min (at 600 W). The solid was filtered, washed with ethanol, dried and recrystallized from ethanol. Yield 68%, m.p. 517 K. Analysis calculated for $C_{26}H_{20}N_6$: C 74.98, H 4.84, N 20.18%; found: C 74.93, H 4.78, N 20.15%.

Crystal data

$C_{26}H_{20}N_6$
$M_r = 416.48$
Triclinic, $P\overline{1}$
$a = 8.4576$ (2) Å
$b = 10.6599$ (3) Å
$c = 12.3642$ (4) Å
$\alpha = 106.9170$ (14)°
$\beta = 98.6240$ (16)°
$\gamma = 100.803$ (2)°
$V = 1022.48$ (5) Å^3
$Z = 2$
$D_x = 1.353$ Mg m^{-3}
Mo Kα radiation
Cell parameters from 4637 reflections
$\theta = 3.1$–27.5°
$\mu = 0.08$ mm^{-1}
$T = 120.0$ (2) K
Block, brown
0.28 × 0.22 × 0.18 mm

Data collection

Nonius KappaCCD diffractometer
φ scans and ω scans with κ offsets
Absorption correction: multi-scan (*DENZO–SMN*; Otwinowski & Minor, 1997)
$T_{min} = 0.977$, $T_{max} = 0.985$
19 117 measured reflections
4637 independent reflections
3732 reflections with $I > 2\sigma(I)$
$R_{int} = 0.050$
$\theta_{max} = 27.5°$
$h = -10 \rightarrow 10$
$k = -13 \rightarrow 13$
$l = -16 \rightarrow 15$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 1.03$
4637 reflections
291 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0603P)^2 + 0.1734P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.25$ e Å^{-3}
$\Delta\rho_{min} = -0.25$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

N1—C1*A*	1.3347 (15)	N52—C53	1.3400 (16)
N1—C8*A*	1.3364 (15)	N52—C51	1.3420 (15)
N2—N3	1.3879 (13)	C1A—C4*A*	1.4206 (15)
N3—C4	1.3139 (15)	C4—C4*A*	1.4375 (16)
N7—C6	1.3147 (15)	C4*A*—C5	1.3953 (17)
N7—N8	1.3896 (13)	C5—C5*A*	1.3954 (16)
N8—C8*A*	1.3717 (15)	C5*A*—C8*A*	1.4240 (15)
C1*A*—N1—C8*A*	110.97 (9)	C5*A*—C5—C4*A*	115.03 (10)
C1*A*—N2—N3	110.74 (9)	C5*A*—C5—C51	122.83 (10)
C1*A*—N2—C21	130.19 (10)	C4*A*—C5—C51	122.07 (10)
N3—N2—C21	119.06 (9)	C5—C5*A*—C8*A*	119.47 (10)
C4—N3—N2	107.25 (9)	C5—C5*A*—C6	135.71 (11)
C6—N7—N8	107.25 (10)	C8*A*—C5*A*—C6	104.67 (10)
C8*A*—N8—N7	110.61 (9)	N7—C6—C5*A*	110.96 (10)
C8*A*—N8—C81	130.72 (10)	N7—C6—C61	119.88 (11)
N7—N8—C81	118.49 (9)	C5A—C6—C61	129.15 (11)
C53—N52—C51	117.14 (10)	N1—C8*A*—N8	126.07 (10)
N1—C1*A*—N2	125.73 (10)	N1—C8*A*—C5*A*	127.41 (10)
N1—C1*A*—C4*A*	127.79 (10)	N8—C8*A*—C5*A*	106.49 (10)
N2—C1*A*—C4*A*	106.48 (10)	N52—C51—C56	122.88 (11)
N3—C4—C4*A*	110.79 (10)	N52—C51—C5	115.80 (10)
N3—C4—C41	120.45 (11)	N52—C53—C54	123.90 (12)
C4*A*—C4—C41	128.74 (11)	C53—C54—C55	118.28 (12)
C5—C4*A*—C1*A*	119.31 (10)	C54—C55—C56	119.28 (12)
C5—C4*A*—C4	135.98 (11)	C55—C56—C51	118.50 (12)
C1*A*—C4*A*—C4	104.71 (10)		
C1*A*—N2—C21—C22	22.81 (18)	C5*A*—C5—C51—C56	70.95 (16)
N3—N2—C21—C22	−156.16 (10)	C4*A*—C5—C51—C56	−112.34 (14)
C1*A*—N2—C21—C26	−158.64 (12)	C8*A*—N8—C81—C82	−3.78 (18)
N3—N2—C21—C26	22.40 (15)	N7—N8—C81—C82	170.86 (10)
C5*A*—C5—C51—N52	−108.40 (13)	C8*A*—N8—C81—C86	177.07 (11)
C4*A*—C5—C51—N52	68.30 (14)	N7—N8—C81—C86	−8.29 (15)

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

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C22—H22···N1	0.95	2.43	3.0029 (16)	119
C82—H82···N1	0.95	2.31	2.9721 (16)	126
C85—H85···N3^{i}	0.95	2.55	3.4922 (18)	173

Symmetry code: (i) $x-1, y-1, z$.

H atoms were treated as riding atoms, with C—H = 0.95 Å (aromatic) and 0.98 Å (methyl), and U_{iso} values of $1.2U_{eq}$(aromatic C) and $1.5U_{eq}$(methyl C).

Data collection: *KappaCCD Server Software* (Nonius, 1997); cell refinement: *DENZO–SMN* (Otwinowski & Minor, 1997); data

reduction: *DENZO–SMN*; program(s) used to solve structure: *SHELXS*97 (Sheldrick, 1997); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *ORTEP*II (Johnson, 1976) and *PLATON* (Spek, 2003); software used to prepare material for publication: *SHELXL*97 and *WordPerfect* macro *PRPKAPPA* (Ferguson, 1999).

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References

Bernstein, J., Davis, R. E., Shimoni, L. & Chang, N.-L. (1995). *Angew. Chem. Int. Ed. Engl.* **34**, 1555–1573.
Ferguson, G. (1999). *PRPKAPPA*. University of Guelph, Canada.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.
Nonius (1997). *KappaCCD Server Software*. Windows 3.11 Version. Nonius BV, Delft, The Netherlands.
Otwinowski, Z. & Minor, W. (1997). *Methods in Enzymology*, Vol. 276, *Macromolecular Crystallography*, Part A, edited by C. W. Carter Jr and R. M. Sweet, pp. 307–326. New York: Academic Press.
Sheldrick, G. M. (1997). *SHELXS*97 and *SHELXL*97. University of Göttingen, Germany.
Spek, A. L. (2003). *J. Appl. Cryst.* **36**, 7–13.